

308      ---      Q200409290003  
Scientific Notebook No. 438: Effect of  
Environment of HE of Ti-7 (01/02/2001  
through 09/19/2003)

# LABORATORY NOTEBOOK

CNWRA/SwRI

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Ti HE  
CLST 20.01402.571  
BROSSIA, X 5797  
Yiming Pan, X 6640

NOTEBOOK NO. \_\_\_\_\_

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Brian K. Derby - *BKD* - BKD  
Steve Clay *STC* KIL  
Jerry Sievert *J.Sievert* JKS  
Roger Dykstra *R.Dykstra* RJD  
Sean Brossia *SB* CBS  
Yiming Pan *Y.Pan* YYP

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TITLE \_\_\_\_\_

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Initial Scientific notebook entry for Titanium Grade 7 Hydrogen Embrittlement Studies.

**Title:** Effect of Environment HE of Ti-7

**Tests Performed by:** Sean Brossia, Steve Clay, other personnel will be identified as they begin work on specific tests.

*Yi-Ming Pan (Div. 20), Jerry Sievert (Div. 18)  
Brian Derby (Div. 18)*

**Objectives:** Determine the conditions under which HE of Ti grades 2, 5, and 7 take place, including critical hydrogen concentration to induce reduction in mechanical properties, effect of orientation, and crack propagation rates if possible

**Equipment:** Slow-strain rate machines, Other specific equipment will be identified and calibrated prior to testing.

**Materials:** Titanium Grades 2,5 and 7 notched slow strain rate specimens procured from Metal Samples

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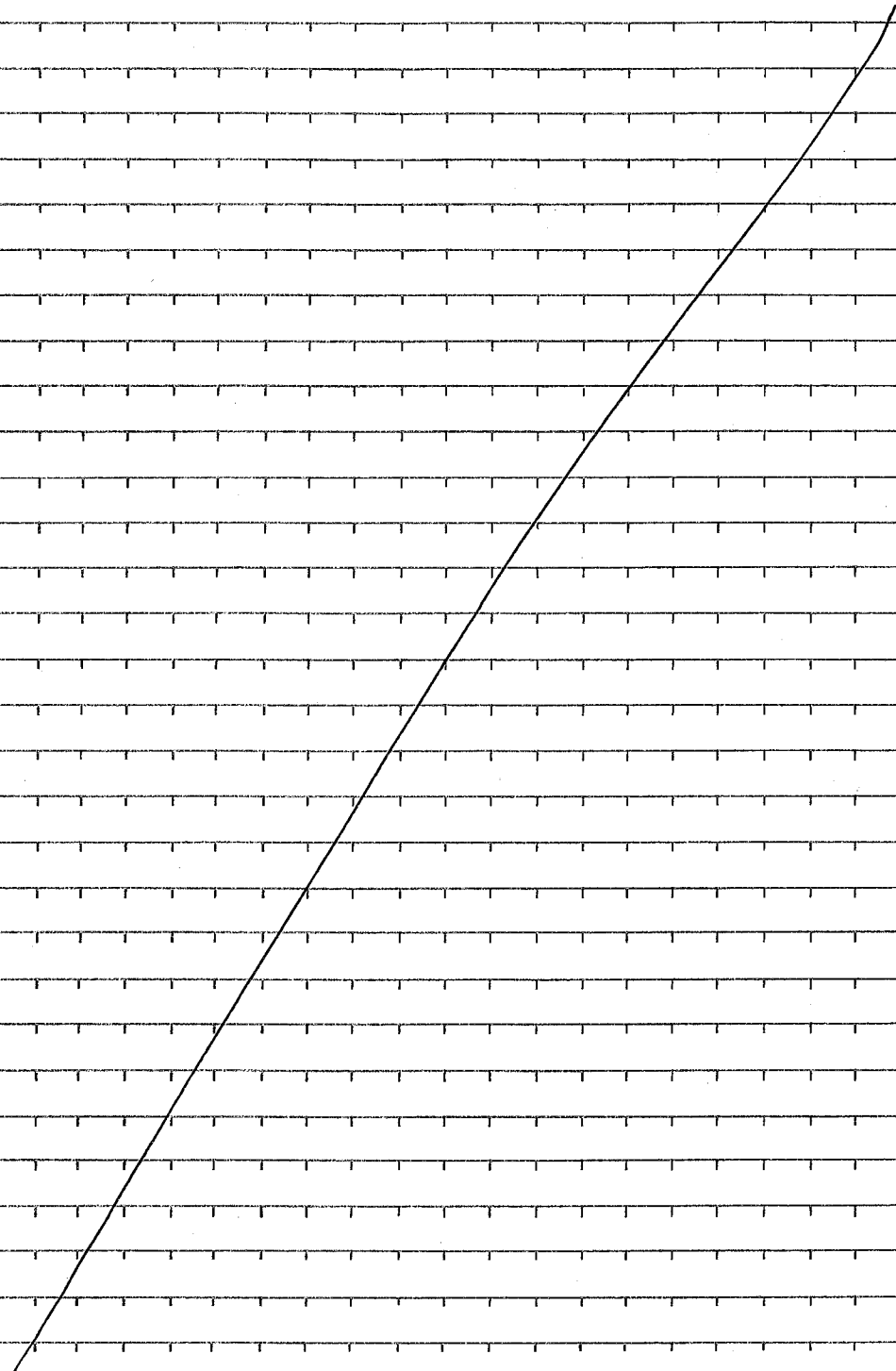
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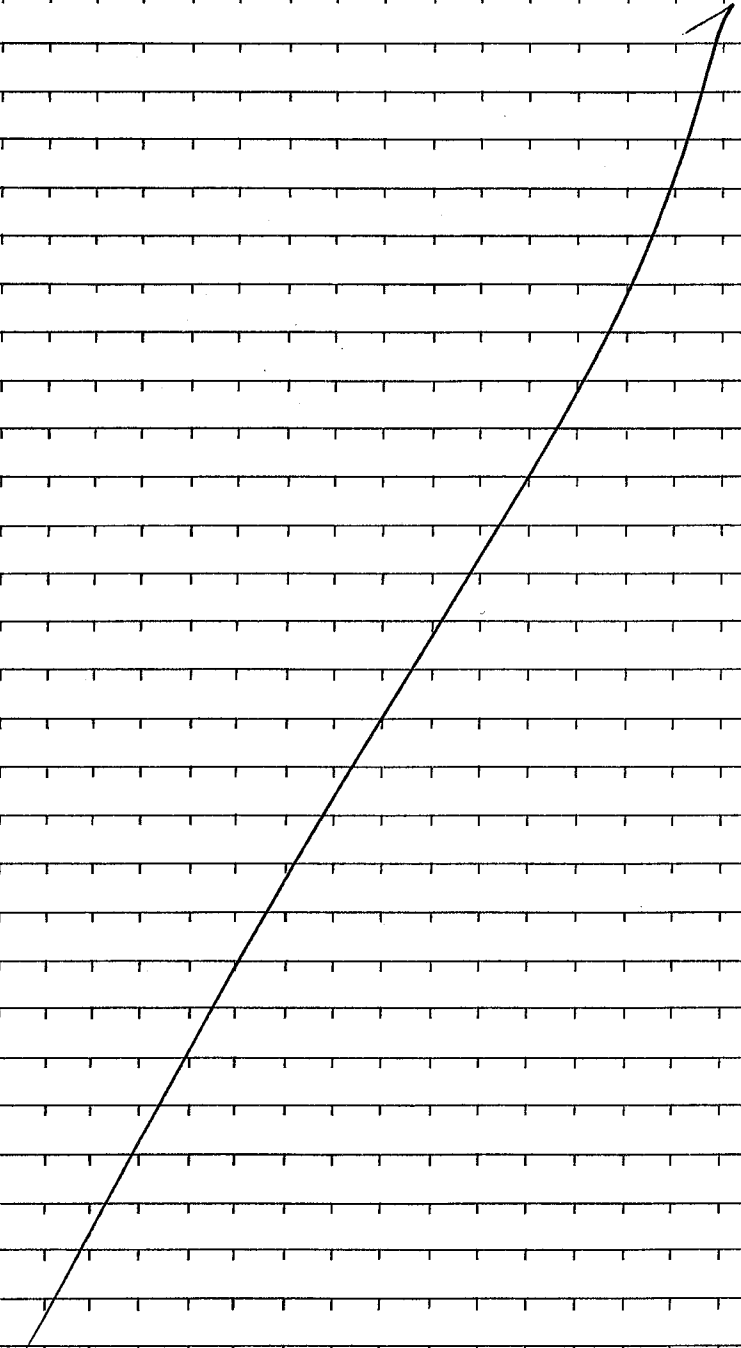
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*Checking Gear Motors for Rotation To Calculate Strain Rate*

Setting	Time for Large Gear #1	Time for Large Gear #2	Average Time	Strain Rate (in/sec)	Strain Rate (mm/sec)
20	279.60	216.20	247.90	6.39E-08	1.62E-06
40	79.30	71.30	75.30	2.10E-07	5.34E-06
60	44.80	41.90	43.35	3.65E-07	9.28E-06
80	31.80	29.90	30.85	5.13E-07	1.30E-05
100	27.90	26.00	26.95	5.88E-07	1.49E-05

Equation  $Y = 1.715529159E-007 * X - 1.449603551E-006$   
 Number of data points used = 5  
 Average X = 60  
 Average Y = 8.84357E-006  
 Residual sum of squares = 1.51641E-012  
 Regression sum of squares = 1.17722E-010  
 Coef of determination, R-squared = 0.987282  
 Residual mean square, sigma-hat-sq'd = 5.05471E-013

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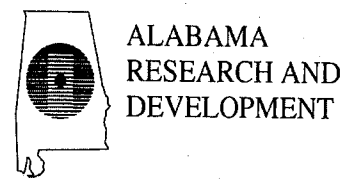
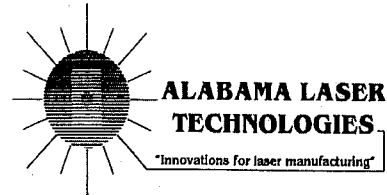
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7	1.00EA	1.00	0.00	CU014829999	
				Description: TIGR2 ARCHIVE SPECIMEN	
8	1.00EA	1.00	0.00	CU014829999	
				Description: TIGR5 ARCHIVE SPECIMEN	
9	1.00EA	1.00	0.00	CU014829999	
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MATERIAL TEST REPORT

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Metal Samples Company  
P.O. Box 8  
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Munford, AL 36268  
Ph. (256)358-4202 Fx. (256)358-4515

Customer: 01482 SOUTHWEST RESEARCH INSTITUTE  
Your PO#: X82483S

Lot No. R951 Mill: Our Order Line No. 1  
Description: TIGR2 .375" X 40.5" X 43.25"  
Item: 018020375 TIGR2 .375"

Chemical Properties:  
C:0.011 Fe:0.111 H:0.001 N:0.016  
O:0.141 Ti:BALANCE

Physical Properties:  
Tensile-PSI:75,000 Elong-%:29.0  
Yield-PSI:50,500 R/A-%:53.3

Lot No. G606 Mill: PRESIDENT TITANIUM Our Order Line No. 3  
Description: TIGR5 5/16"THK  
Item: 018070312 TIGR5 .312"

Chemical Properties:  
Al:6.180 C:0.030 Fe:0.140 H:0.0123  
N:0.007 O:0.186 Ti:BALANCE V:4.050  
Y:<0.0005

Physical Properties:  
Tensile-PSI:146,000 Elong-%:10.9  
Yield-PSI:131,000 R/A-%:25.4

Lot No. P586 Mill: TIMET Our Order Line No. 5  
Description: TIGR7 1.00" X 24.00" X 48.00"

Chemical Properties:  
C:0.009 Fe:0.115 H:50 PPM N:0.007  
O:0.140 Pd:0.155 Ti:BALANCE

Physical Properties:  
Tensile-PSI:79,000 Elong-%:25.0  
Yield-PSI:54,000 R/A-%:45.0  
Condition:ANLD

We certify that the Material Test Report is correct to the best of our knowledge and that the material supplied meets your required P.O. specifications.

THANK YOU, Quality Control Dept.

*[Signature]*

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### Galvanic Corrosion Scan

Objective: To obtain E<sub>applied</sub> Potential for further Tests

Specimens: Ti7 Polishes To 600 Grit Finish  
516 cylinders Polishes To 600 Grit Finish

No Specimen weights Taken

Solution: 1 M Cl<sup>-</sup> + 0.1 M F<sup>-</sup>  
58.45 g NaCl Lot# 006924  
4.201 g NaF Lot# 991559  
+ DI water To 1000mls

Potentiostat: PC4/750

Reference: Fisher 13-620-52 SN# 0089777

Temperature: 95°C Hg Thermometer SN# C96-649 Cal 6/29/01

Solution Deaerated with 99.999% N<sub>2</sub>

Data saved As ti7\_fe\_galv1.dta

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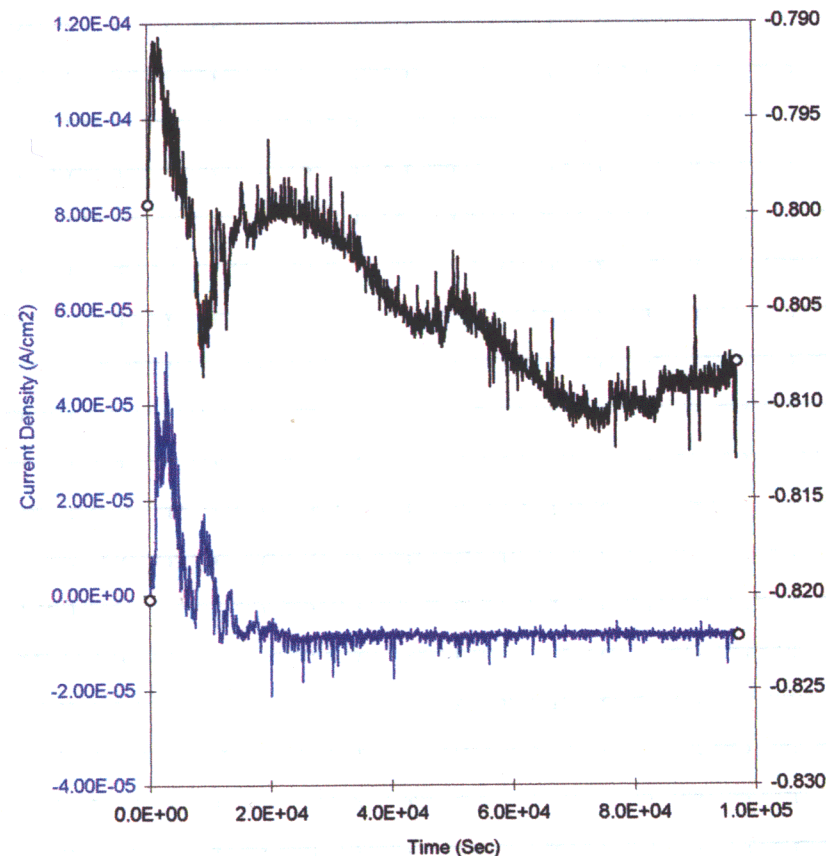
*B. K. D.*

5/29/01

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checked all at time (signed)

### Galvanic Corrosion Scan 'ti7\_fe\_galv1.dta' 8/27/2001-9:54:13



Pstat: PC4/750  
 Run Time: 259200 S  
 Sample Time: 60 S  
 Cur Limit: 100 mA  
 Eoc: -0.78703 V  
 Area: 1 cm<sup>2</sup>  
 Electrode: 7.87 gm/cm<sup>3</sup>, 27.92 g/equiv  
 Delay: OFF  
 IR Comp.: OFF

NOTES

ANALYSIS

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Testing Plan for the Initial Set of Ti Slow Strain Rate Tests

No. of Tests: 6 (3 materials and 2 orientations)

Materials: Ti Grades 2, 5, 7

Orientations: Parallel or perpendicular to rolling direction

Specimen Type: Notched tensile specimens

Test Conditions: Deaerated solution — 1M NaCl + 0.1M NaF  
 Temperature — 95 °C  
 Extension rate —  $1 \times 10^{-5}$  mm/s (depending on the gear motors)  
 Open circuit

Data Monitoring: Load  
 Temperature  
 Potential (not available under open-circuit conditions)  
 Current (not available under open-circuit conditions)  
 pH (initial/final)

After Test Analysis: Elongation  
 Failure time  
 Fractograph  
 XRD

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Test Solutions:

<sup>0.1 M NaF</sup>  
Solution I - 1.0M NaCl + ~~1.0~~ M NaF, deaerated with N<sub>2</sub> (Test set #1)  
Solution II - 1.0M NaCl, deaerated with N<sub>2</sub> (Test set #2)

Data Monitoring:

Load vs. time

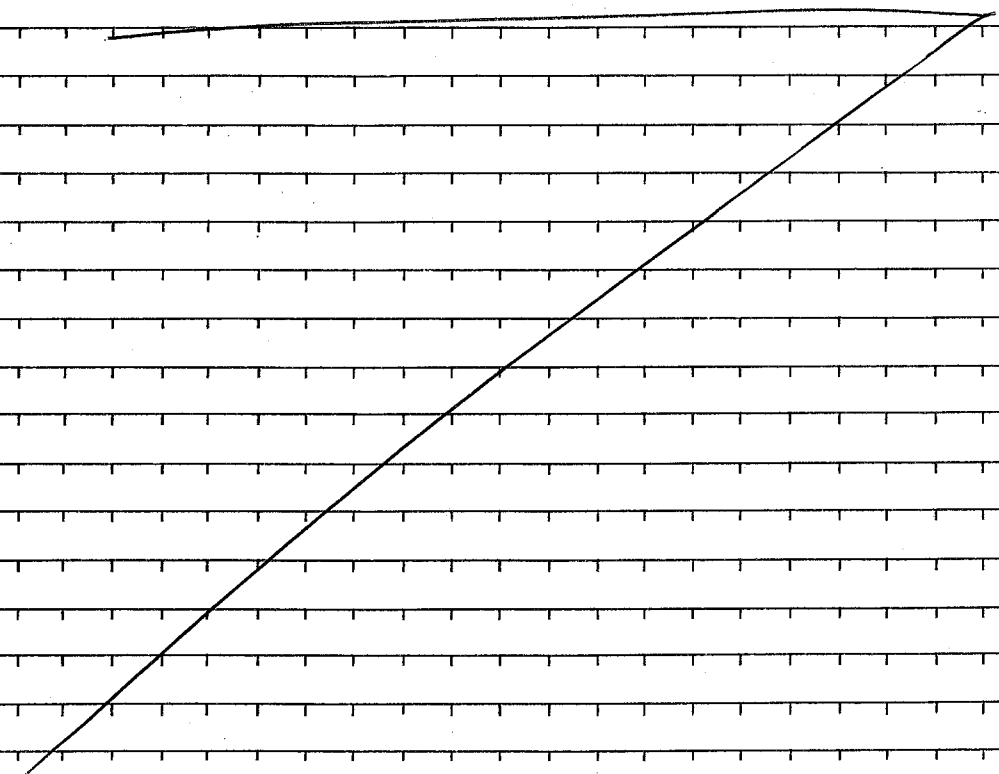
Displacement vs. time

Potential vs. time

Current vs. time

} only on one specimen and periodical check on the rest of the specimens\*

\* Note: Because all channels in the ESC multichannel potentiostat share to same common ground, potential can not be monitored for all six channels using one reference electrode. Thus, monitoring of potential and current vs. time will be only on Ti Grade 7 specimen.



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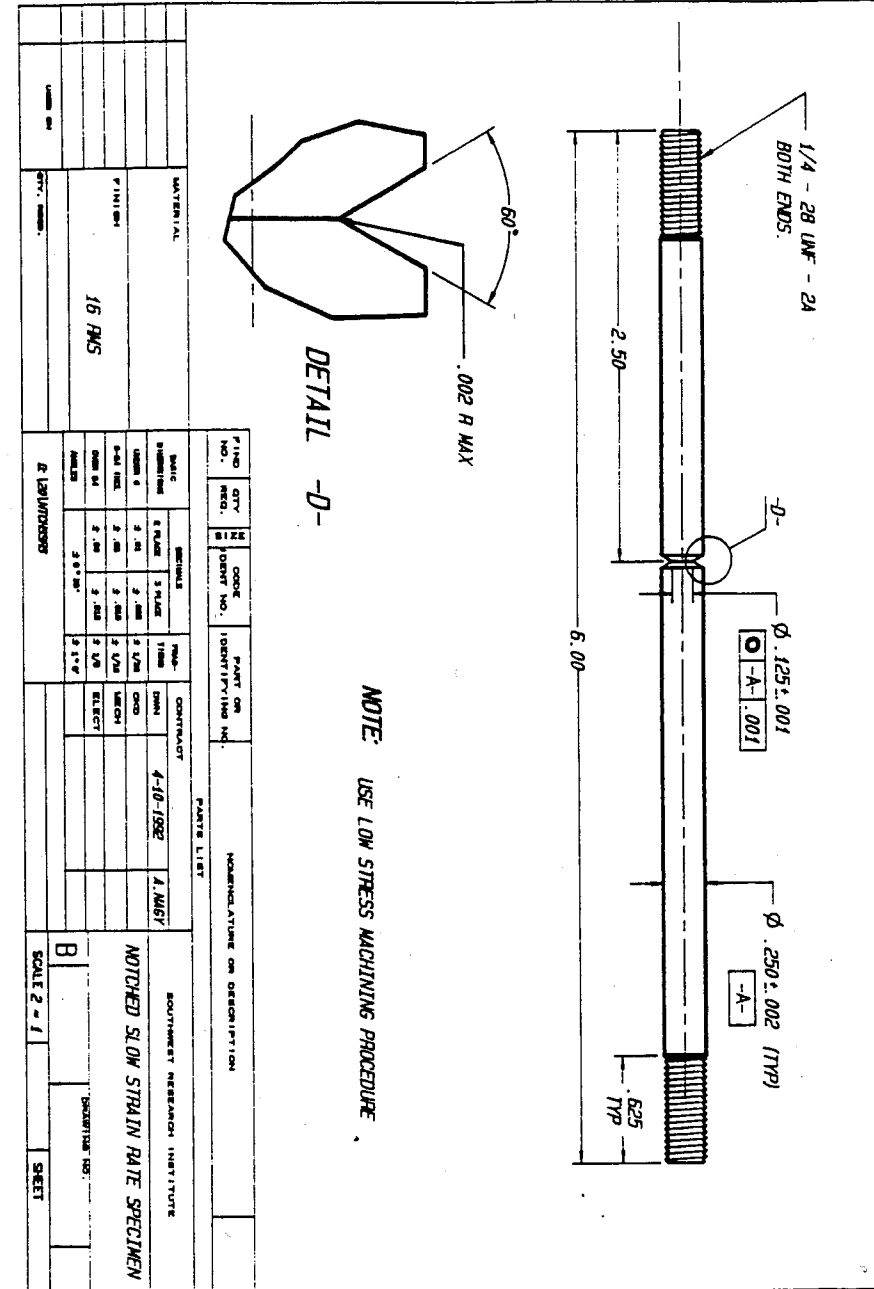
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6/27/02

From Page No. \_\_\_\_\_

Dimensions



Drawing Number: 20.1402.571.012

Originator: *[Signature]*  
Darrell Dunn

Date: 9/8/2000

Reviewed by: *[Signature]*  
Narasi Sridhar

Date: 9/8/2000

QA Approval: *[Signature]*  
Bruce Mabrito

Date: 9/8/2000

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Load Cells Channels 1-6

Equipment used:  
 Morehouse Proving Ring s/p 3668C-709 Due 1/15/09  
 Keithley 617 Electrometer s/p 537418 Due 3/11/03

Date: 6/25/02 Frame: SS5 Load Cell: 1

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.8	-0.2mV	0	9.7	2.5mV	0	0.0012	0
600	59.9	0.4968	591.07	59.0	0.4996	586.85	0.4982	586.46
1200	109.6	1.0041	1178.36	109.6	1.0056	1179.54	1.0048	1178.95
1800	159.2	1.5109	1766.29	159.4	1.5109	1769.86	1.5109	1768.08
2400	209.3	2.017	2361.08	210.1	2.016	2371.83	2.016	2366.46
3000	259.1	2.521	2954.38	260.9	2.520	2976.83	2.520	2965.60
3600	309.4	3.024	3554.61	309.4	3.025	3555.80	3.024	3555.20
4000	342.2	3.360	3947.02	342.1	3.361	3947.02	3.360	3947.02

SHUNT CAL: 5.552 ✓

Date: 6/25/02 Frame: SS5 Load Cell: \*2

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.9	4mV	0	9.9	2mV	0	0.0012	0
600	59.0	0.5639	579.48	59.0	0.5659	579.48	0.5649	579.48
1200	109.4	1.1385	1174.81	109.3	1.1402	1173.63	1.1394	1174.22
1800	160.6	1.7126	1781.50	160.8	1.7136	1783.88	1.7131	1782.69
2400	210.6	2.285	2375.40	210.4	2.285	2373.02	2.285	2374.21
3000	261.1	2.857	2976.83	260.2	2.856	2966.07	2.856	2971.45
3600	310.2	3.428	3563.02	310.4	3.427	3565.41	3.428	3564.22
4000	344.0	3.809	3967.32	343.5	3.808	3961.36	3.808	3964.34

SHUNT CAL: 6.280V

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Date: 6/25/02 Frame: SS5 Load Cell: \*3

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.9	9mV	0	9.8	2.59mV	0	0.0042	0
600	59.0	0.894	579.48	59.9	0.893	591.07	0.8914	585.28
1200	109.4	1.863	1174.81	109.4	1.884	1175.99	1.8874	1175.40
1800	160.1	2.806	1775.56	160.0	2.822	1775.56	2.814	1775.54
2400	210.9	3.746	2378.06	210.8	3.749	2378.96	3.748	2378.96
3000	260.6	4.68	2970.85	260.8	4.68	2974.44	4.68	2972.64
3600	310.6	5.62	3567.80	310.1	5.62	3563.02	5.62	3565.41
4000	343.8	6.56	3964.94	343.8	6.56	3966.13	6.56	3965.54

SHUNT CAL: 5.422 ✓

Date: 6/25/02 Frame: SS5 Load Cell: \*4

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.2	0.65mV	0	9.2	5.74mV	0	0.0032	0
600	62.0	1.5240	623	62.4	1.5341	627.72	1.5290	625.36
1200	110.1	3.035	1191.43	111.1	3.0416	1203.25	3.0398	1197.34
1800	160.6	4.546	1789.81	161.7	4.5535	1802.89	4.5494	1796.35
2400	210.4	6.057	2381.34	211.4	6.0631	2393.23	6.0555	2387.28
3000	260.1	7.568	2973.24	261.3	7.5739	2987.57	7.5634	2980.40
3600	311.9	9.079	3565.14	311.3	9.085	3584.52	9.079	3588.10
4000	344.0	10.59	3975.69	345.0	10.594	3987.64	10.59	3981.66

SHUNT CAL: 5.342 ✓

Date: 6/26/02 Frame: SS5 Load Cell: \*5

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.4	-5mV	0	9.7	4.1mV	0	0.019	0
600	61.6	1.5155	615.90	62.0	1.5243	619.09	1.5199	616.50
1200	109.1	3.032	1177.18	110.2	3.0478	1186.70	3.0420	1181.94
1800	160.8	4.549	1789.81	161.6	4.5649	1795.76	4.5538	1792.78
2400	210.6	6.066	2381.34	211.6	6.081	2389.66	6.075	2385.50
3000	260.5	7.583	2975.63	261.5	7.598	2984.00	7.584	2979.82
3600	310.7	9.098	3574.97	311.9	9.113	3585.72	9.104	3580.34
4000	343.6	10.615	3968.52	344.9	10.631	3980.47	10.626	3974.50

SHUNT CAL: 5.411V

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From Page No. 15

Typical  
Statistical  
Plot of  
Load Cell  
Interaction

Nonlinear Regression

[Variables]  
x = col(1)  
y = col(2)  
'Automatic Initial Parameter Estimate Functions  
F(q)=ape(x,y,1,0,1)  
[Parameters]  
y0 = F(0)[1] "Auto {{previous: -2.11528}}  
a = F(0)[2] "Auto {{previous: 1175.79}}  
[Equation]  
f=y0+a\*x  
fit f to y  
[Constraints]  
[Options]  
tolerance=0.000100  
stepsize=100  
iterations=100

R = 0.99999717 Rsqr = 0.99999434 Adj Rsqr = 0.99999340

Standard Error of Estimate = 3.6315

	Coefficient	Std. Error	t	P
y0	-2.1153	2.3673	-0.8935	0.4060
a	1175.7858	1.1418	1029.7838	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	13985229.5582	13985229.5582	1060454.7501	<0.0001
Residual	6	79.1277	13.1880		
Total	7	13985308.6859	1997901.2408		

PRESS = 119.1921

Durbin-Watson Statistic = 1.6695

Normality Test: Passed (P = 0.6084)

Constant Variance Test: Passed (P = 0.7053)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	-0.7043	0.7043	0.1940	0.2557	0.2347
2	583.6612	2.7988	0.7707	0.9070	0.8913
3	1179.3143	-0.3643	-0.1003	-0.1107	-0.1011
4	1774.3794	-6.2994	-1.7347	-1.8600	-2.6096
5	2368.2688	-1.8088	-0.4981	-0.5348	-0.5002
6	2960.8649	4.7351	1.3039	1.4442	1.6322
7	3553.4609	1.7391	0.4789	0.5673	0.5324
8	3948.5249	-1.5049	-0.4144	-0.5279	-0.4935

Influence Diagnostics:

Row	Cook's Dist	Leverage	DFFITS
1	0.0241	0.4245	0.2016
2	0.1583	0.2779	0.5529
3	0.0013	0.1787	-0.0472
4	0.2591	0.1303	-1.0100
5	0.0218	0.1324	-0.1954
6	0.2365	0.1849	0.7773
7	0.0649	0.2875	0.3382
8	0.0868	0.3838	-0.3895

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	-0.7043	-6.4941	5.0854	-11.3101	9.9014
2	583.6612	578.9768	588.3456	573.6161	593.7063
3	1179.3143	1175.5578	1183.0707	1169.6669	1188.9616
4	1774.3794	1771.1722	1777.5867	1764.9323	1783.8265
5	2368.2688	2365.0352	2371.5025	2358.8127	2377.7249
6	2960.8649	2957.0444	2964.6853	2951.1923	2970.5374
7	3553.4609	3548.6963	3558.2254	3543.3781	3563.5436
8	3948.5249	3943.0197	3954.0301	3938.0717	3958.9781

Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

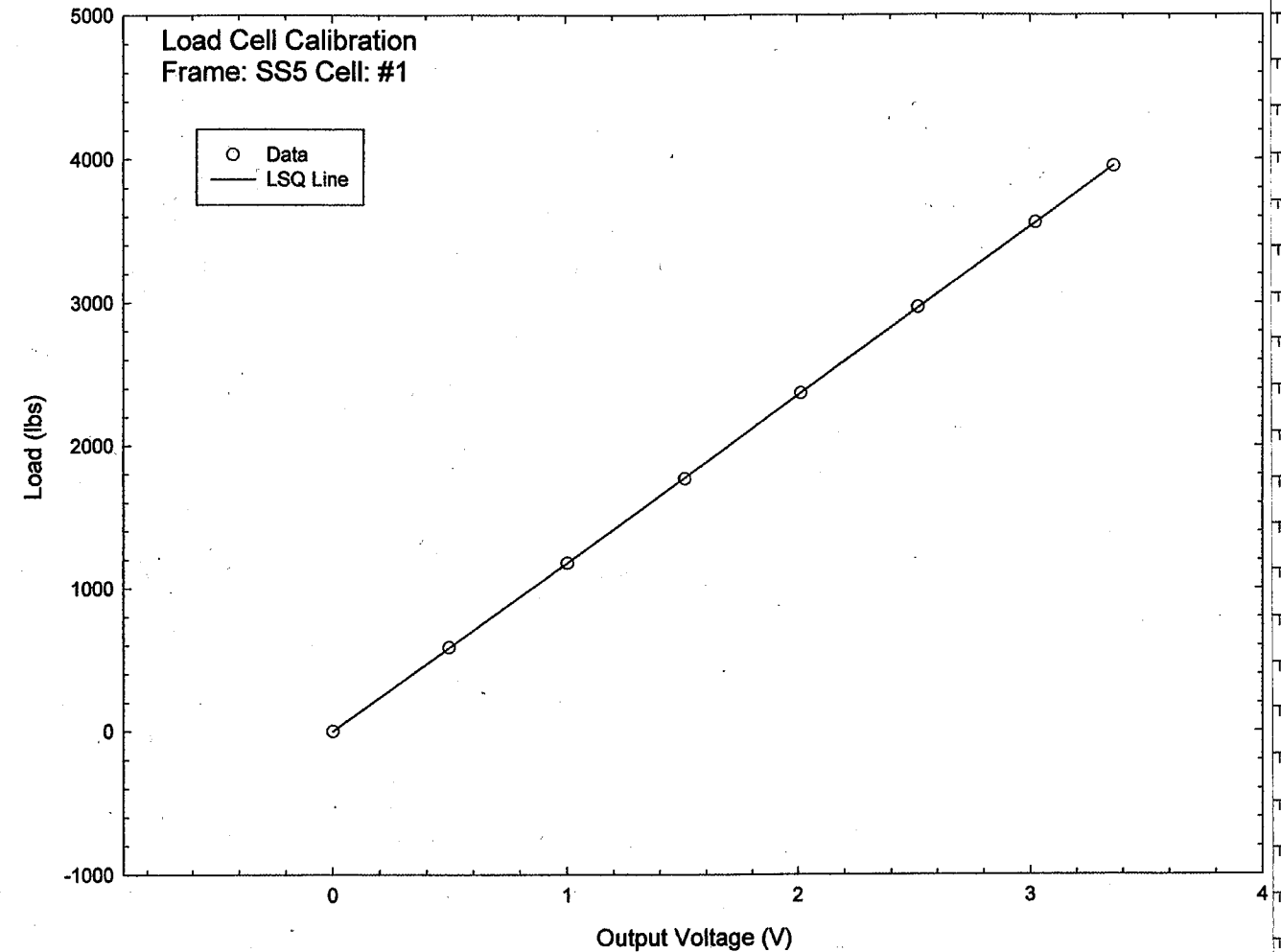
Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

From Page No. 16



Typical Load Cell Plot

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by Steve Os

7-30-02

To Page No. \_\_\_\_\_

From Page No. 15

Date: 6/26/02 Frame: SS5 Load Cell: #6

Dead Weight	Ring Reading	Output Voltage	Load	Ring Reading	Output Voltage	Load	Average Voltage	Average Load
0	9.7	0.1 mV	0	9.6	1.5 mV	0	0.0008	0
600	61.1	1.5095	606.44	60.6	1.5051	601.71	0.5073	604.09
1200	110.4	1.6019	1189.07	110.4	1.6024	1190.25	1.0022	1189.66
1800	161.7	1.5022	1796.95	161.9	1.5023	1800.52	1.5022	1798.74
2400	211.7	2.002	2390.85	211.8	2.001	2393.23	2.002	2392.04
3000	261.6	2.500	2985.19	261.8	2.500	2988.77	2.500	2986.98
3600	311.8	2.997	3584.52	311.0	2.997	3576.17	2.997	3580.34
4000	344.4	3.329	3974.49	344.8	3.328	3980.47	3.328	3977.48

SHUNT CAL: 5.369V

Load Cell Cals:  $f=y_0+A*X$

LC	$y_0$	A
1	-2.1153	1175.789
2	-6.4951	1042.225
3	-10.1332	1208.32
4	1.8534	1178.357
5	-5.0143	1192.815
6	-2.5485	1195.831

Cal Factors per volt used in software

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by [Signature]

7-3-02

From Page No. 18

LVDT

Equipment used:

Kentley 617 Electrometer \$0 537418 Due 3/11/03  
Gauge Blocks (Date Below) Due 1/7/03

```

*****
*                               *
* Carl Zeiss, Incorporated      *
*                               *
* 7008 Northland Drive        *
* Minneapolis, Mn. 55428      *
* Tel. (612) 533-9990        *
* Fax (612) 533-4903        *
*                               *
*****
Program name:
Date: 01/07/102              Time: 14:10          Program Number: 0
*****                          Page: 1
*                               *
* Description                  Value           Tolerance      *
*                               *
* DISTANCE Plane (Feature # 1) to Point (Feature # 2) 1/16 RHW X379A
*                               *
*                               *
* DISTANCE Plane (Feature # 3) to Point (Feature # 4) 1/16 RHW X369A
*                               *
*                               *
* DISTANCE Plane (Feature # 5) to Point (Feature # 6) 1/25 RHW Y242A
*                               *
*                               *
* DISTANCE Plane (Feature # 7) to Point (Feature # 8) 1/25 RHW Y455A
*                               *
*                               *
* DISTANCE Plane (Feature # 9) to Point (Feature # 10) 1/50 RHW 694A
*                               *
*                               *
* DISTANCE Plane (Feature # 11) to Point (Feature # 12) 1/100 RHW Y191B
*                               *
*                               *
*****

```

Bldg # 162  
Temp. 70°  
Humidity 30%  
Small CMM X529

JAN -7 2002

LVDT	Model S/N	VL7A L4904600	Part Number 060-3618-01	7/3/2002 S. Clay		
Gauge Blk	Travel Inch	Volt Run 1	Volt Run 2	Volt Run 3	Cal Factor	
0.0000	1.0000	10.000	10.000	10.000	10.000	0.1
0.0625	0.9375	9.366	9.367	9.367	9.367	0.10008897
0.1250	0.8750	8.739	8.739	8.741	8.740	0.10011823
0.1875	0.8125	8.106	8.107	8.112	8.108	0.10020555
0.2500	0.7500	7.486	7.485	7.486	7.486	0.10019148
0.3750	0.6250	6.240	6.242	6.243	6.242	0.10013351
0.5000	0.5000	5.001	5.001	5.004	5.002	0.09996002
0.6250	0.3750	3.755	3.753	3.757	3.755	0.09986684
0.7500	0.2500	2.506	2.503	2.508	2.506	0.09977385
0.8750	0.1250	1.249	1.247	1.251	1.249	0.10008006
1.0000	0.0000	0.002	0.002	0.002	0.002	0

Cal Factor Avg. = 0.10004185

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by [Signature]

7-3-02



From Page No. \_\_\_\_\_

Solution 1M NaCl → 116.88g Fisher 010488  
 1M NaF → 8.398g Fisher 896405  
 2 liters HPDI PH = 8.398 ~~7/18/02~~

Two baths required for 2.8 liter volume.

Specimen position in autoclave head

Position #6 Grade 7 Per ○	Position #1 Grade 2 Per ○
Position #5 Grade 5 Per ○	Position #2 Grade 5 Per ○
Position #4 Grade 2 Per ○	Position #3 Grade 7 Per ○

Specimens placed in Auto clamp with 2.8 liters Solution  
 Deaerated w/ N<sub>2</sub> for 24 hours while bringing solution  
 temperature to 95°C

Extension rate set to  $1 \times 10^{-6}$  in/sec

Open circuit potential of Grade 7 Per recorded  
 Load + Deflection vs Time recorded all Channels

Reference probe is Silver Silver Chloride w/ .1 M Potassium Chloride

Data Storage filenames Spec1, Spec2, Spec3, Spec4, Spec5, Spec6  
 New filenames: Ti2PER, Ti5PER, Ti7PER  
 Ti2PAR, Ti5PAR, Ti7PAR

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

7-9-02

Solution 1M NaCl → 116.88g Fisher 010488  
 2 liters HPDI PH = 6.165 ~~7/18/02~~

Two 2 liter batches

Specimen position (See page 20)

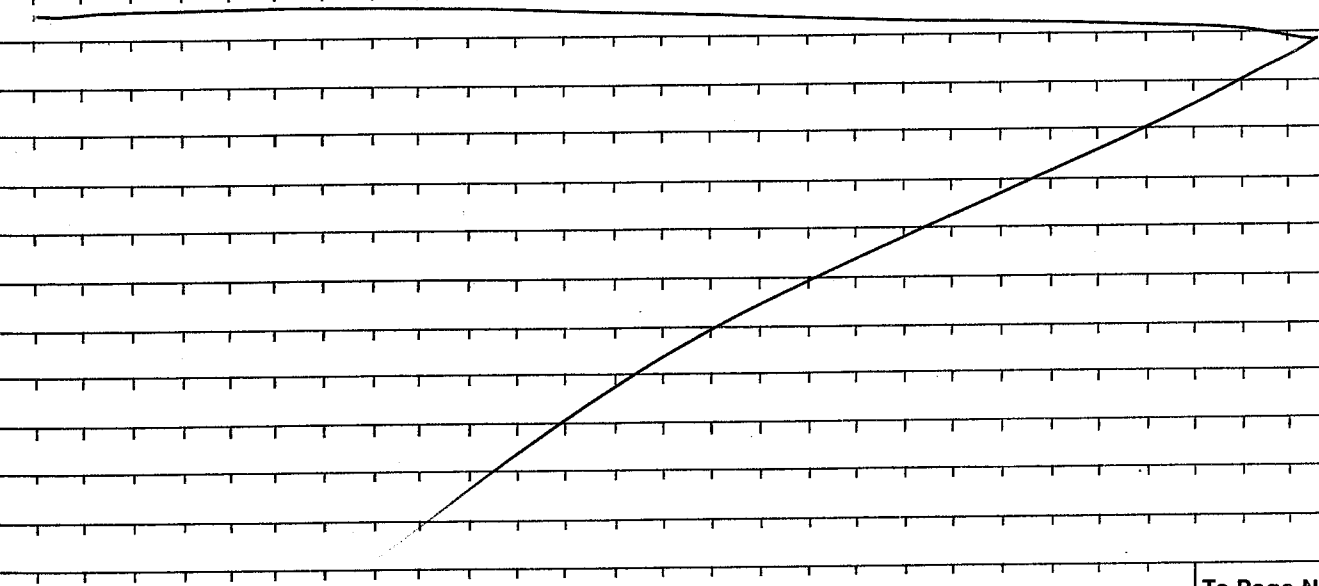
Specimens placed in Auto clamp with 2.8 liters Solution  
 Deaerated for 24 hours while bringing temperature to 95°C

Extension rate set to  $1 \times 10^{-6}$  in/sec

Open circuit potential of Grade 7 Per recorded  
 Load + Deflection vs Time recorded all channels

Reference probe is Silver Silver Chloride w/ .1 M Potassium Chloride

Data Storage filenames  
 Ti2PER2.1, Ti5PAR2.2, Ti7PER2.3,  
 Ti2PAR2.4, Ti5PER2.5, Ti7PAR2.6



To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

7-16-02

From Page No. \_\_\_\_\_

Solution is N/A, test conducted in Air

Specimen position (see page 20)

Specimens placed in Auto Clave, no solution was added. Test was in Air Heated up to 45°C

Extension rate was  $1 \times 10^{-6}$  in/sec

Open circuit potential of grade 7 Per recorded hand; Deflection vs time recorded on All channels

Reference probe is Silver Silver chloride w/ .1M Potassium chloride

Data filenames

Ti2 per 3.1, Ti5 per 3.2, Ti7 per 3.3

Ti2 per 3.4, Ti5 per 3.5, Ti7 per 3.6

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

*Roger J. Dijkstra*

7/29/02

From Page No. \_\_\_\_\_

The reduction in area of the slow strain rate specimens was calculated by measuring the area of the fracture surface dividing by the initial cross section area of the specimens (0.125 in. in diameter). The data are recorded below.

SSRT Specimens Reduction in Area

File Name	Specimen ID	Test Condition	Area(mm <sup>2</sup> )	RA(%)
Ti2PAR1	TiGr2-1(L)	1M NaCl+0.1M NaF	6.21	21.5
Ti2PER1	TiGr2-1(T)	1M NaCl+0.1M NaF	5.03	36.4
Ti5PAR1	TiGr5-1(L)	1M NaCl+0.1M NaF	6.67	15.7
Ti7PAR1	TiGr7-1(L)	1M NaCl+0.1M NaF	5.86	25.9
Ti7PER1	TiGr7-1(T)	1M NaCl+0.1M NaF	5.11	35.4
Ti2PAR2	TiGr2-2(L)	1M NaCl	5.68	28.2
Ti2PER2	TiGr2-2(T)	1M NaCl	5.34	32.6
Ti5PAR2	TiGr5-2(L)	1M NaCl	6.36	19.6
Ti5PER2	TiGr5-2(T)	1M NaCl	6.49	18.0
Ti7PAR2	TiGr7-2(L)	1M NaCl	5.29	33.1
Ti7PER2	TiGr7-2(T)	1M NaCl	5.13	35.2
Ti2PAR3	TiGr2-3(L)	Air	5.82	26.5
Ti2PER3	TiGr2-3(T)	Air	5.66	28.5
Ti5PAR3	TiGr5-3(L)	Air	6.39	19.2
Ti5PER3	TiGr5-3(T)	Air	6.84	13.6
Ti7PAR3	TiGr7-3(L)	Air	5.85	26.1
Ti7PER3	TiGr7-3(T)	Air	6.05	23.5

RA(%) = 100 - (Area/((0.125\*25.4)<sup>2</sup>\*3.14/4))\*100

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

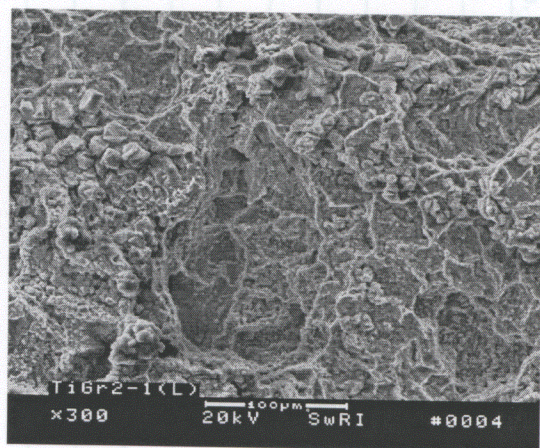
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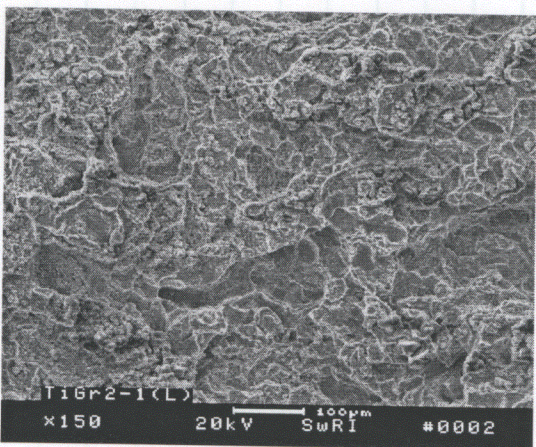
*Y. Pan*

7/29/02

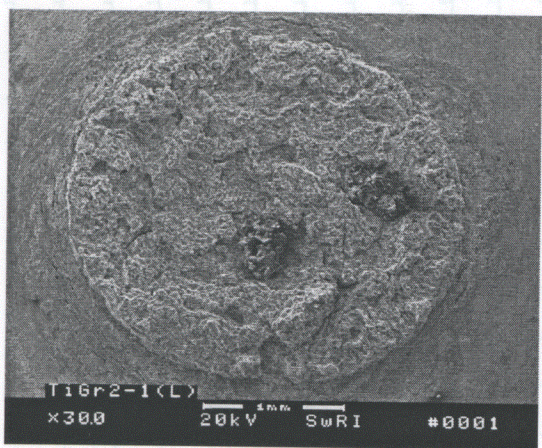
From Page No. \_\_\_\_\_



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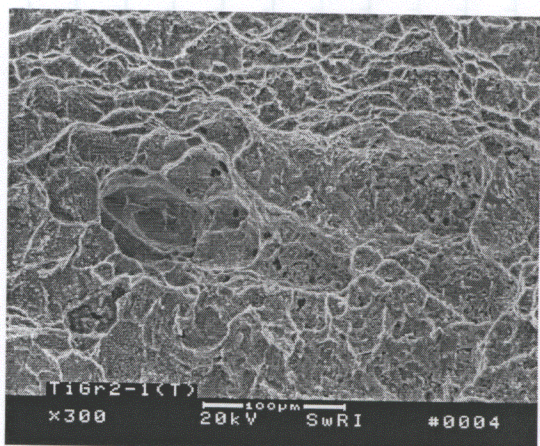


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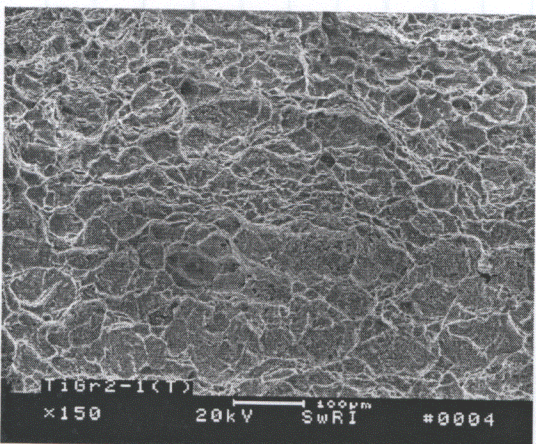


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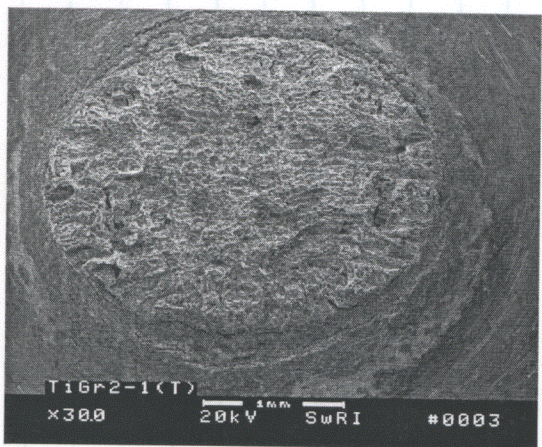
30x



300x



150x



TiGr2-1(T)

30x

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

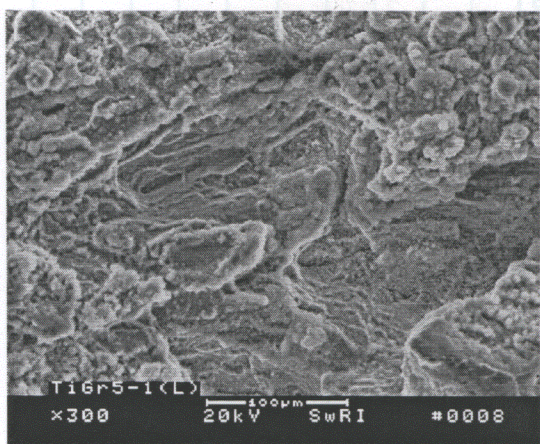
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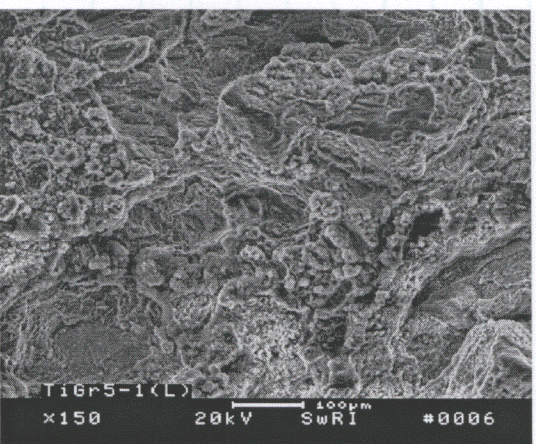
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No. \_\_\_\_\_

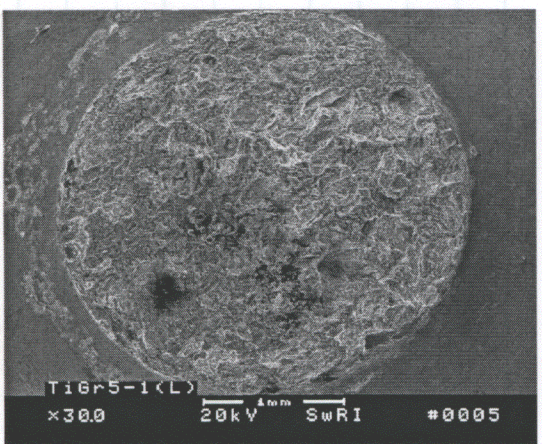
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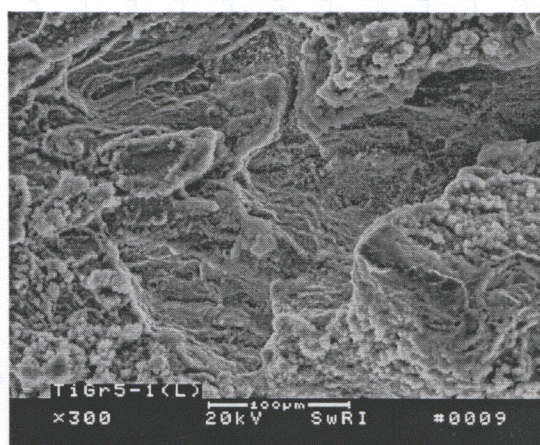


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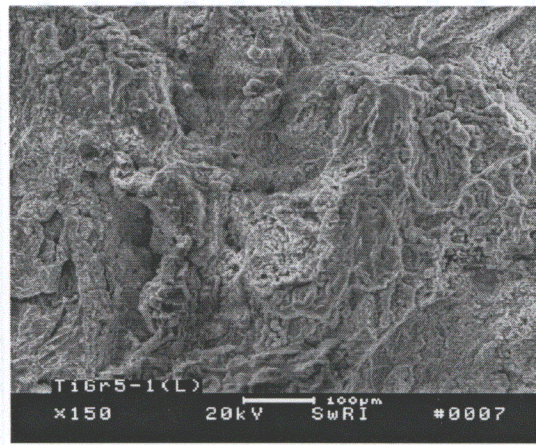


TiGr5-1(L)

30x



300x



150x

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

Date \_\_\_\_\_

No. \_\_\_\_\_



From Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

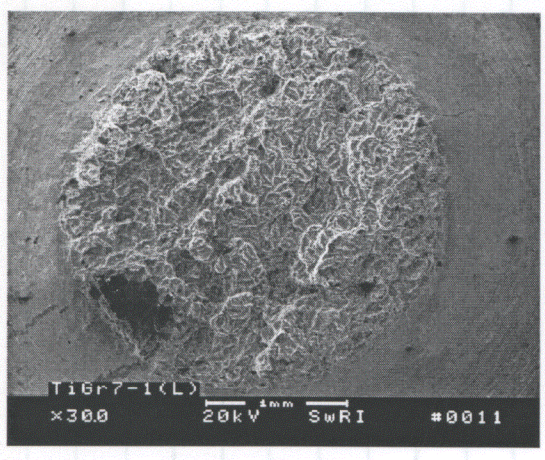
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Invented by \_\_\_\_\_

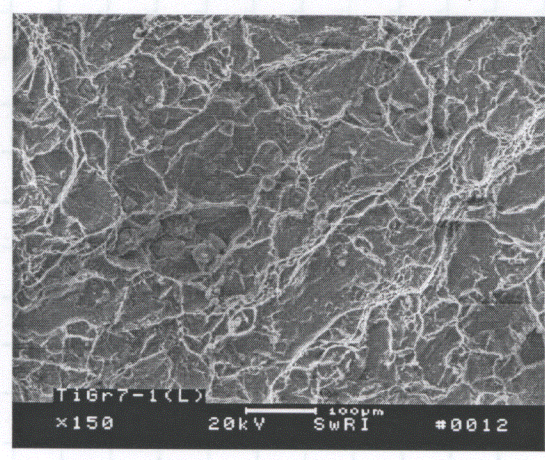
Date \_\_\_\_\_

Recorded by *M. J. Pa*

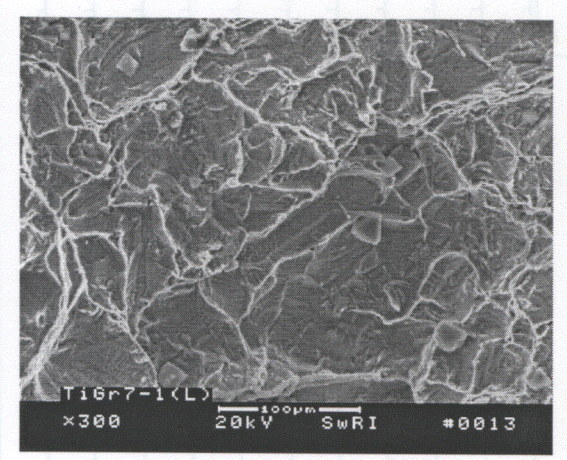
*7/30/02*



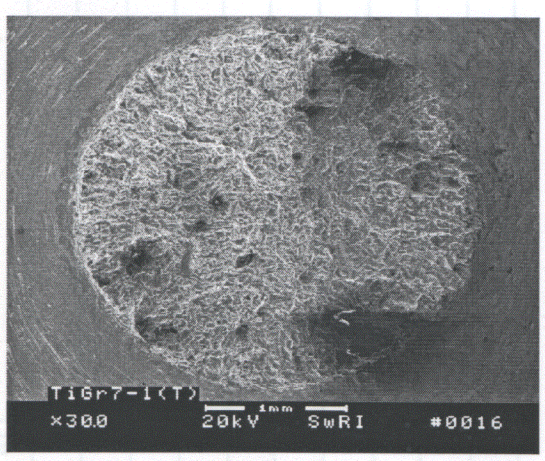
TiGr7-1(L) 30x



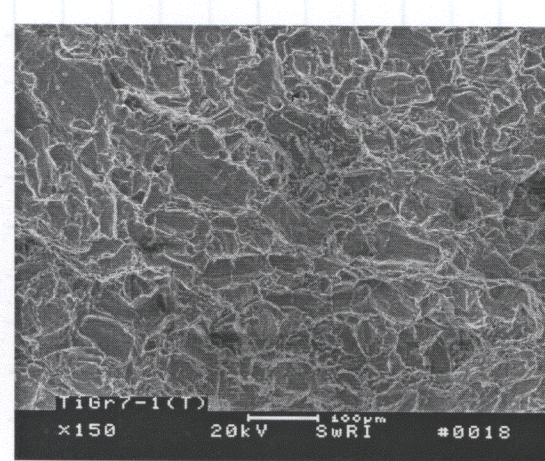
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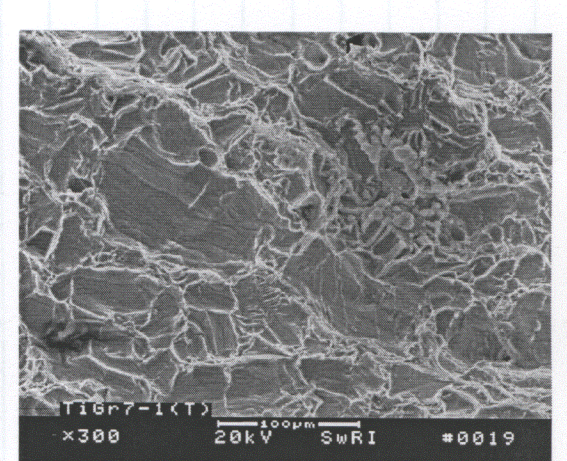
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TiGr7-1(T) 30x



150x



300x

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

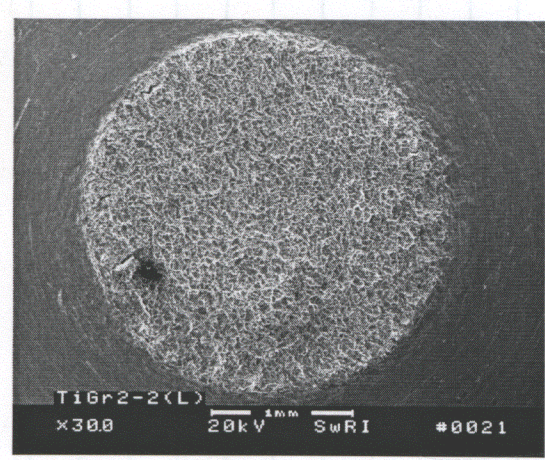
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Date \_\_\_\_\_

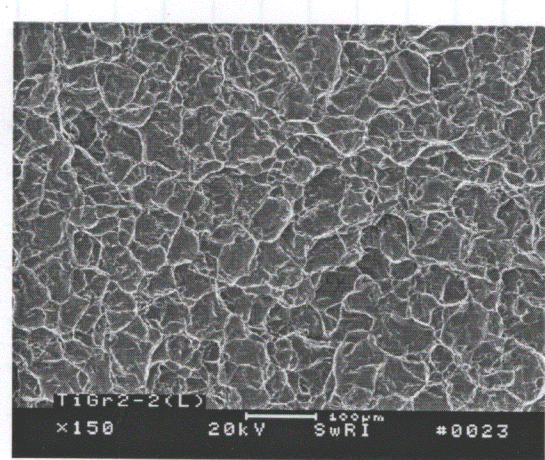
Recorded by *M. J. Pa*

*7/30/02*

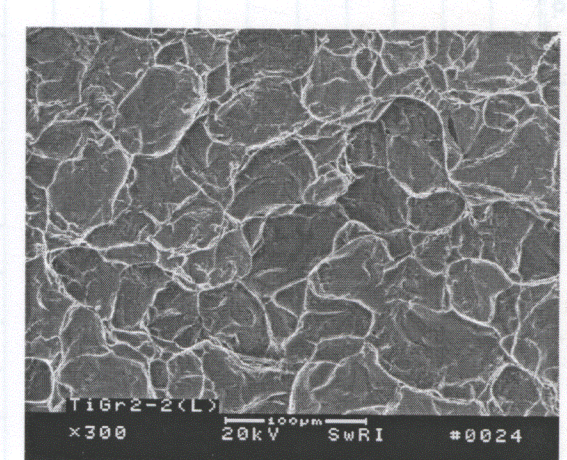
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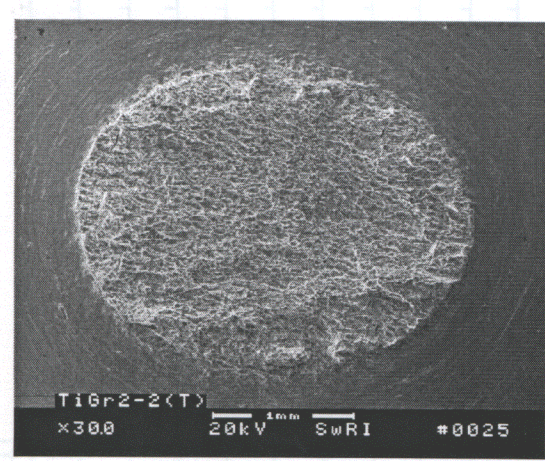
TiGr2-2(L) 30x



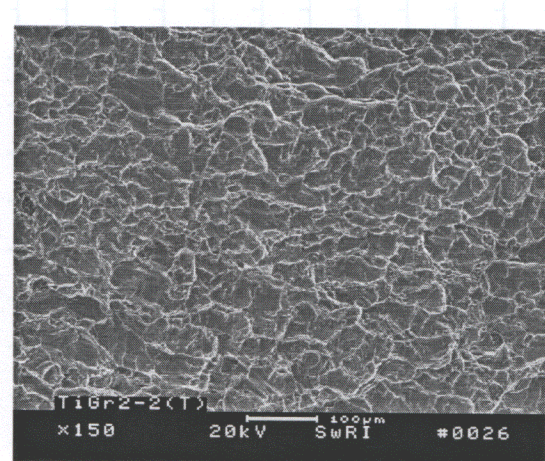
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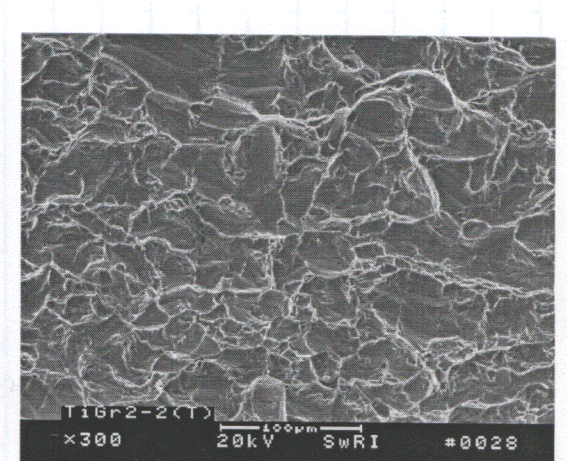
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TiGr2-2(T) 30x

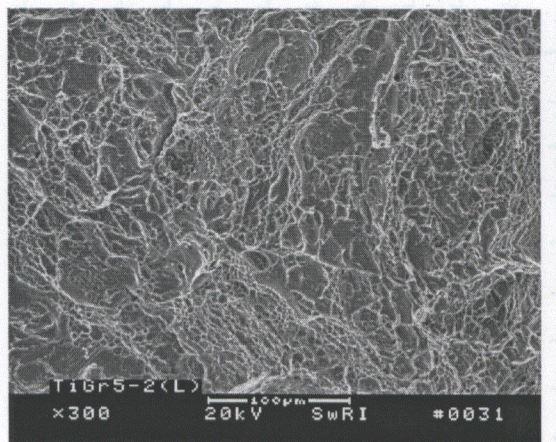


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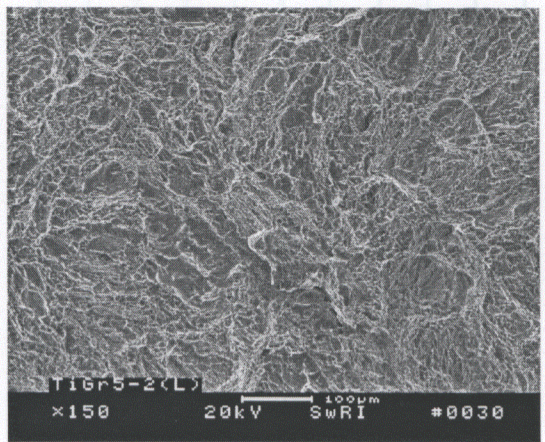


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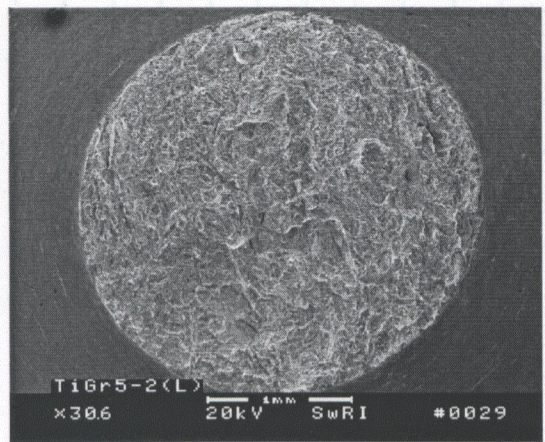
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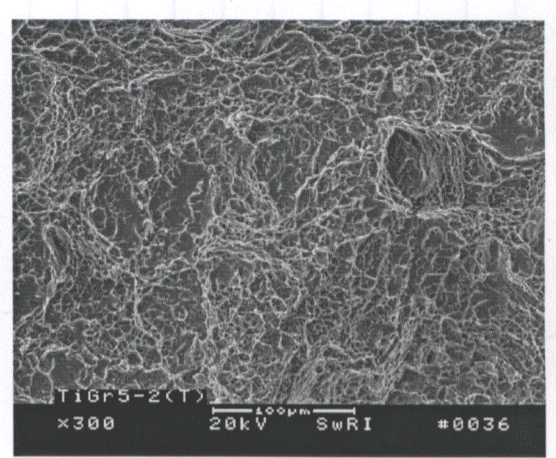
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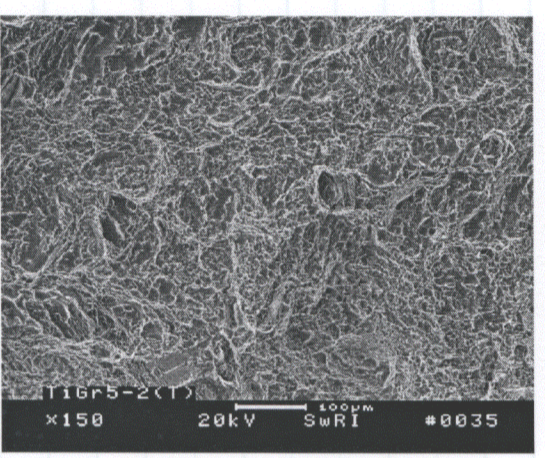
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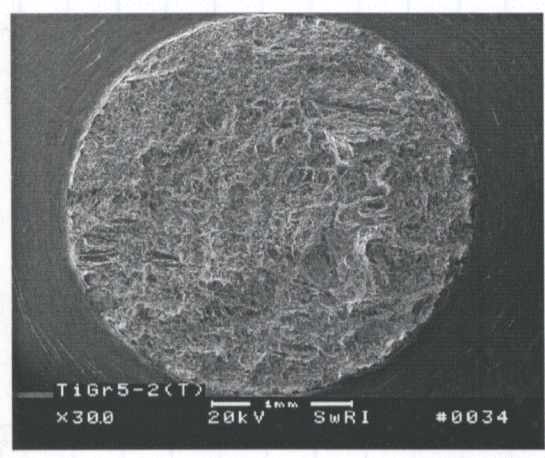
30x



300x



150x



30x

Witnessed & Understood by me, \_\_\_\_\_

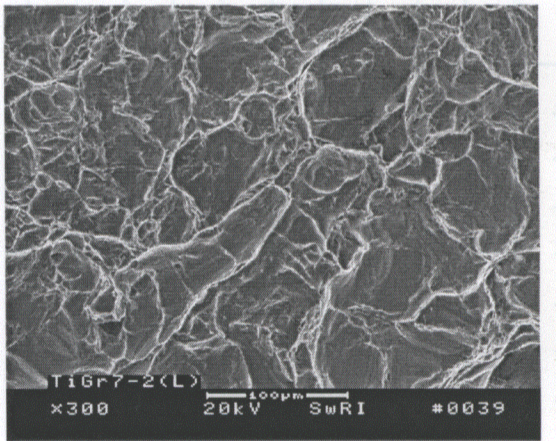
Date \_\_\_\_\_

Invented by \_\_\_\_\_

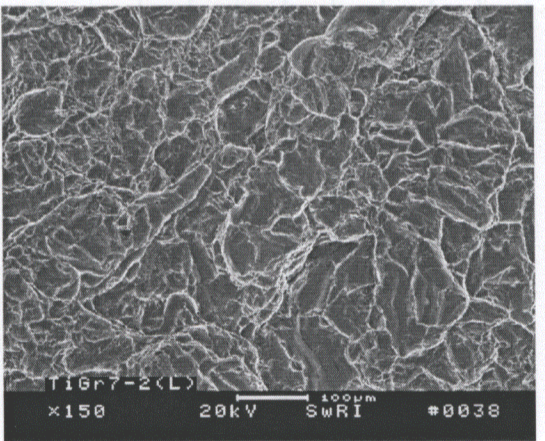
Recorded by \_\_\_\_\_

Date 7/30/02

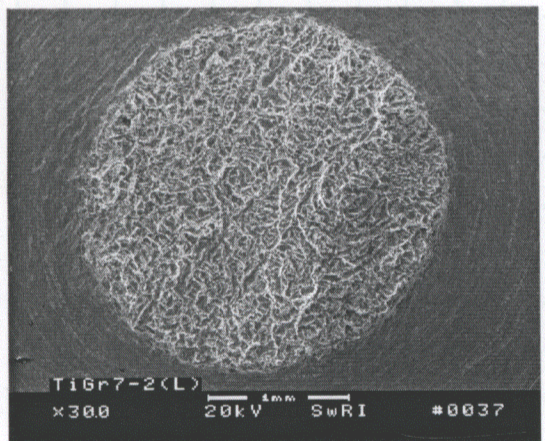
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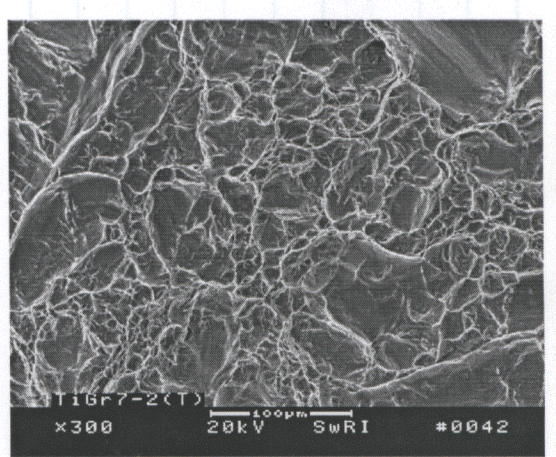
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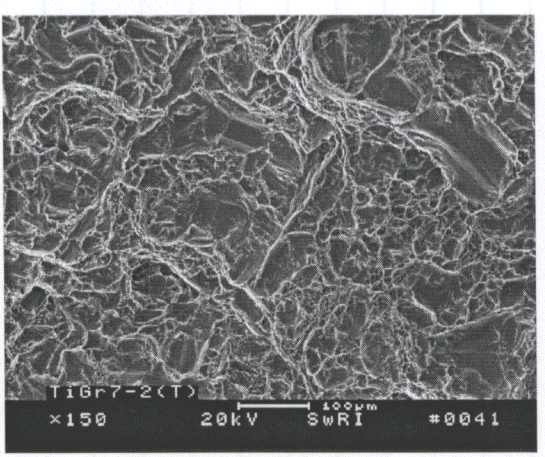
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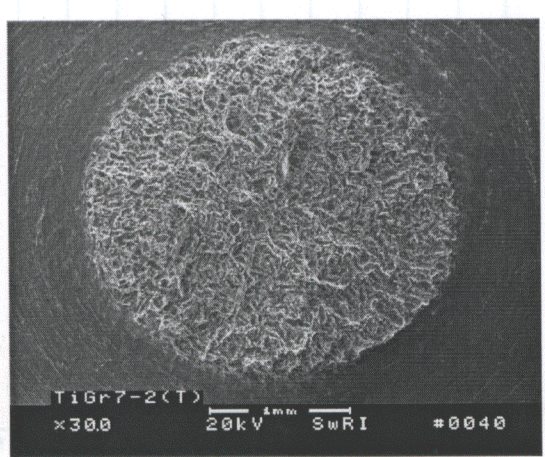
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300x



150x



30x

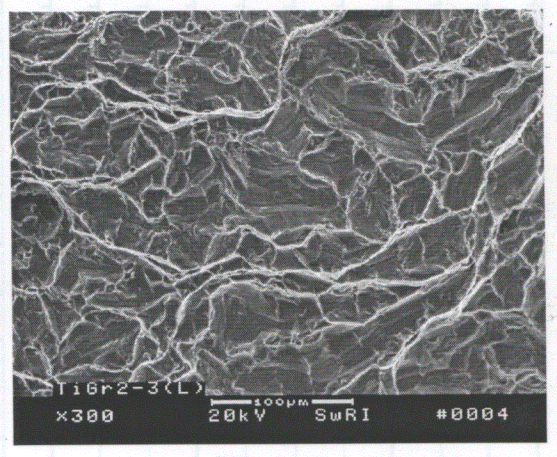
Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

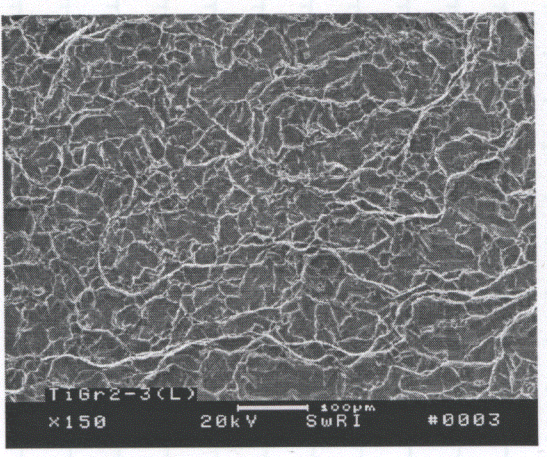
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Recorded by \_\_\_\_\_

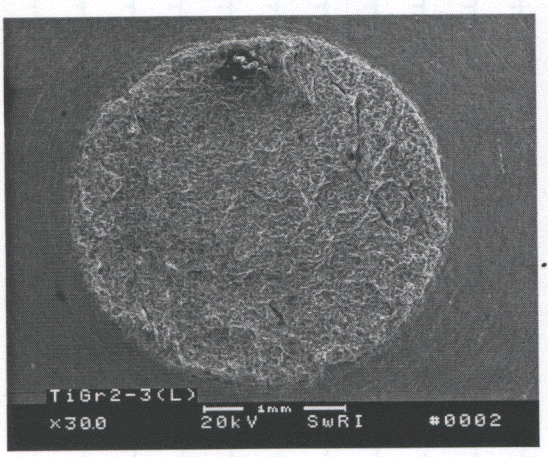
Date 7/30/02



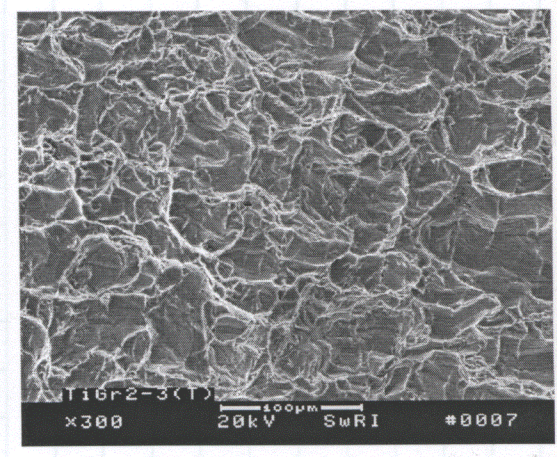
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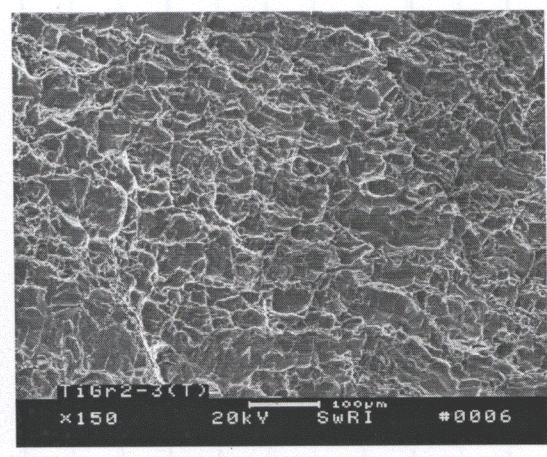
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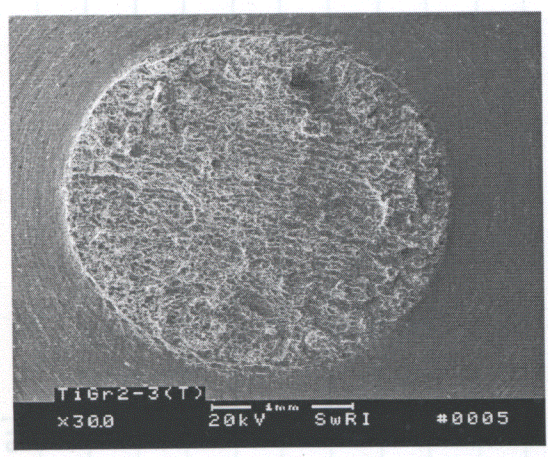
TiGr2-3(L) 30x



300x



150x



TiGr2-3(T) 30x

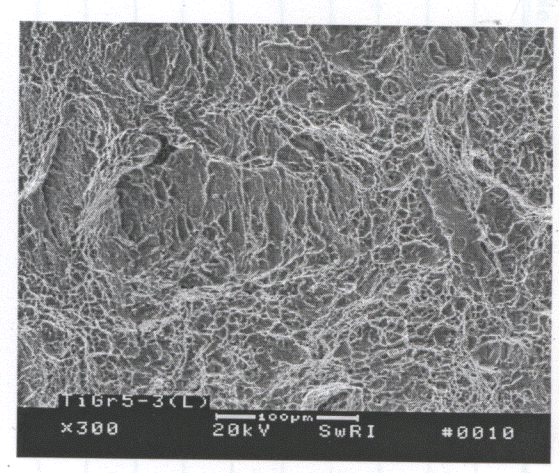
Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Recorded by *M.P. Ra*

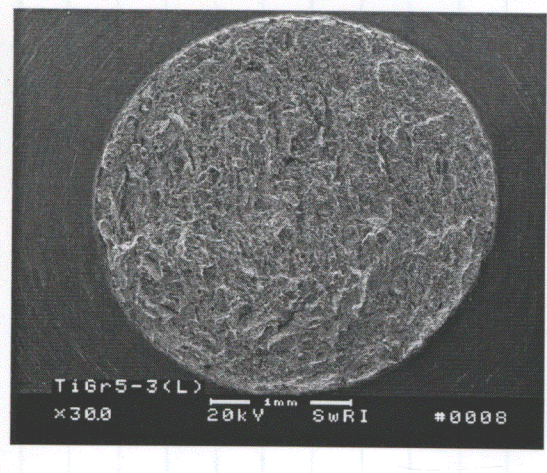
Date *7/30/02*



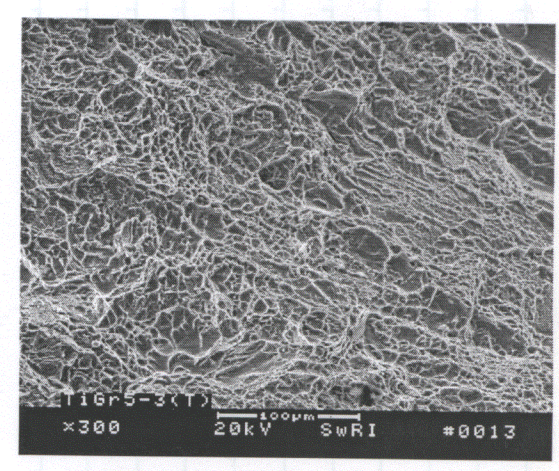
300x



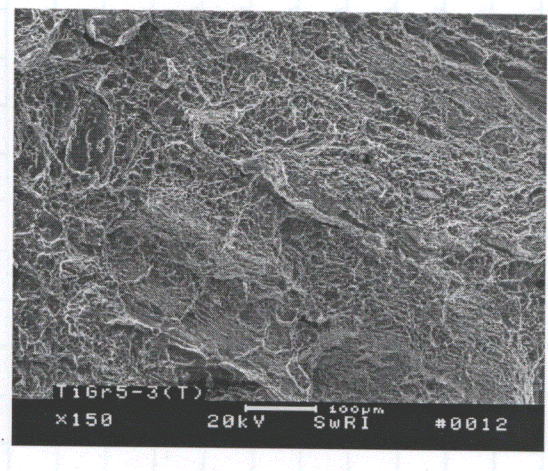
150x



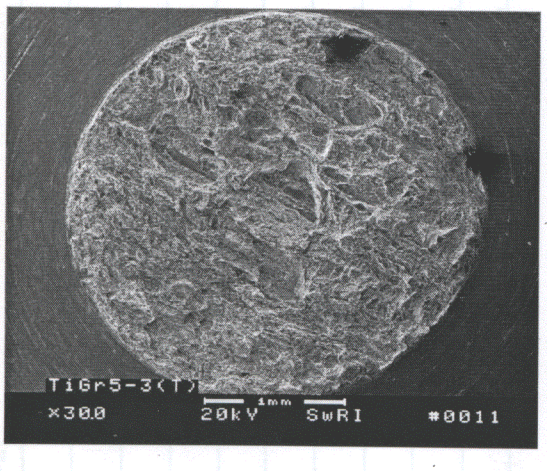
TiGr5-3(L) 30x



300x



150x



TiGr5-3(T) 30x

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

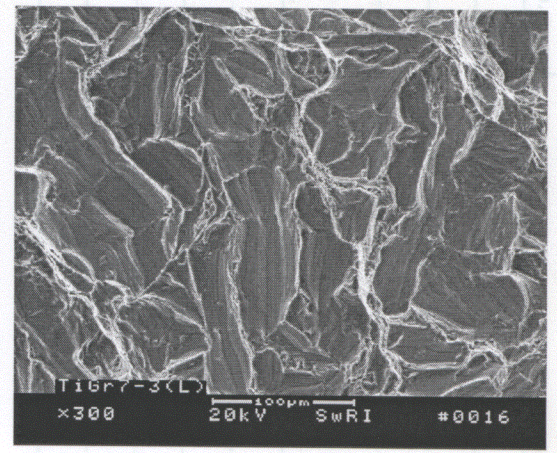
Invented by \_\_\_\_\_

Recorded by *M.P. Ra*

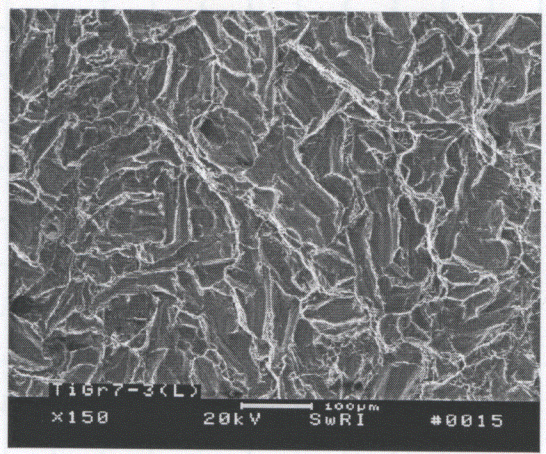
Date *7/30/02*

No. \_\_\_\_\_

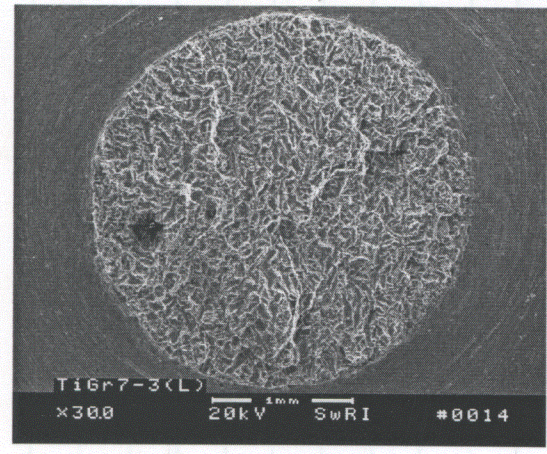
From Page No. \_\_\_\_\_



300x

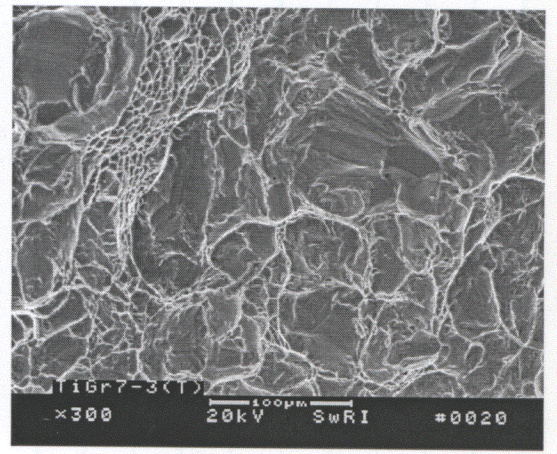


150x

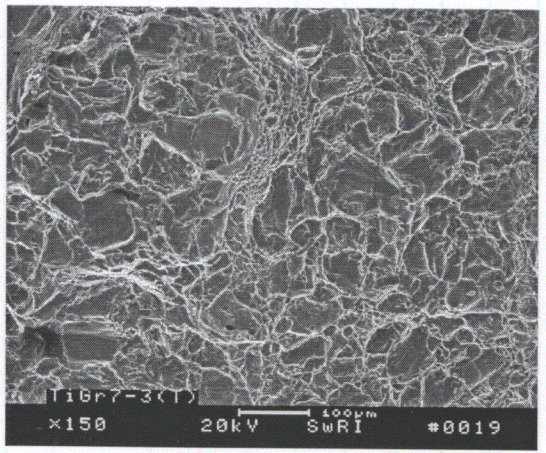


30x

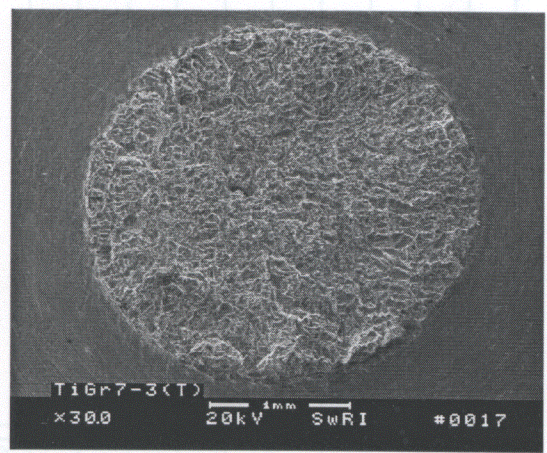
TiGr7-3(L)



300x



150x



30x

TiGr7-3(T)

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Recorded by \_\_\_\_\_

Date \_\_\_\_\_

7/30/02

No. \_\_\_\_\_

From Page No. \_\_\_\_\_

Solution 1M NaCl → 116.90g Fisher 02081A  
0.1M NaF → 8.40g Fisher 99155A  
2 liters HPDI pH = 8.971 <sup>imp 10/8/02</sup>

1M NaCl → 58.45g Fisher 02081A  
0.1M NaF → 4.20g Fisher 99155A  
1 liter HPDI pH = 8.863 <sup>imp 10/8/02</sup>

Specimen position (see page 20)

Specimens placed in Autoclave with 2.8 liters of solution. Deaired for 24 hours while bringing up temperature to 95°C

Extension rate set to  $1 \times 10^{-7}$  in/sec

Open circuit potential of grade 7 PAR recorded hourly & Deflection vs Time recorded all channels

Reference probe is Silver Silver Chloride w/.1M Potassium chloride

Data Storage filenames  
TI2 per 4.1, TIS per 4.2, TI7 per 4.3  
TI2 per 4.4, TIS per 4.5, TI7 per 4.6

Copies sent to QA Records

*[Signature]* 9/11/02

To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>Roger J. Rykstra</i>	8-28-02

From Page No. \_\_\_\_\_

FY2003 Augmented Work  
Title: Effect of Galvanic Coupling on Hydrogen-Induced Cracking of Titanium Grade 7

Description:  
Environmentally assisted cracking arising from hydrogen absorption has been considered a possible failure mode for the titanium drip shield. Hydrogen-induced cracking of Titanium Grade 7 needs to be evaluated by considering the effect of galvanic coupling to carbon and stainless steels on enhancing hydrogen uptake and absorption. This augmented work is aimed at determining the hydrogen-induced cracking susceptibility of Titanium Grade 7, as well as Grades 2 and 5, in fluoride-containing solution as a result of galvanic coupling in comparison to solutions without fluoride. Preliminary scoping experiments are planned with galvanically coupled specimens using a six-specimen slow strain rate test machine. Notched slow strain rate specimens will be galvanically coupled to a cathode made of carbon steel or stainless steel. All slow strain rate tests will be conducted in 1.0 M NaCl solutions with and without the addition of 0.1 M NaF at 95 °C [203 °F]. The fracture surfaces of all specimens will be examined by scanning electron microscopy. Selected specimens will be further analyzed to verify hydride formation and to measure hydrogen concentrations.

Cost Estimate (Outside Div. 20):  
Total Cost: \$16,000

- Slow strain rate testing (\$13,000)
  - Test machine modification
  - Preparation of galvanically coupled specimens
  - Conducting slow strain rate tests
- Metallurgical analysis (\$1,000)
- Hydrogen concentration analysis (\$2,000)

Test Set #1 for Ti SSRT on the Effect of Galvanic Coupling

No. of Tests: 2 (one each of two materials)

Materials: Ti Grades 5 and 7

Orientation: Parallel to rolling direction

Specimen Type: Notched Ti tensile specimens coupled with cast iron

Test Conditions: Deserated solution C 1M NaCl + 0.1M NaF  
Temperature C 95 EC  
Extension rate C  $1 \times 10^{-7}$  in/s  
Open circuit

Data Monitoring: Load vs. Time  
Deflection vs. Time  
Potential and Current vs. Time

Measuring both the initial and final pHs

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date *7/16/03*  
Recorded by *Jerry Schmit*

From Page No. \_\_\_\_\_

TEST SOLUTION: 1M NaCl + 0.1M NaF

NaCl 58.45g FISHER LOT 905502  
NaF 4.2g FISHER LOT 896405

DI WATER TO MAKE 1L PH = 8.809

NaCl 116.9g FISHER LOT 905522  
NaF 8.4g ALDRICH LOT 0601KQ

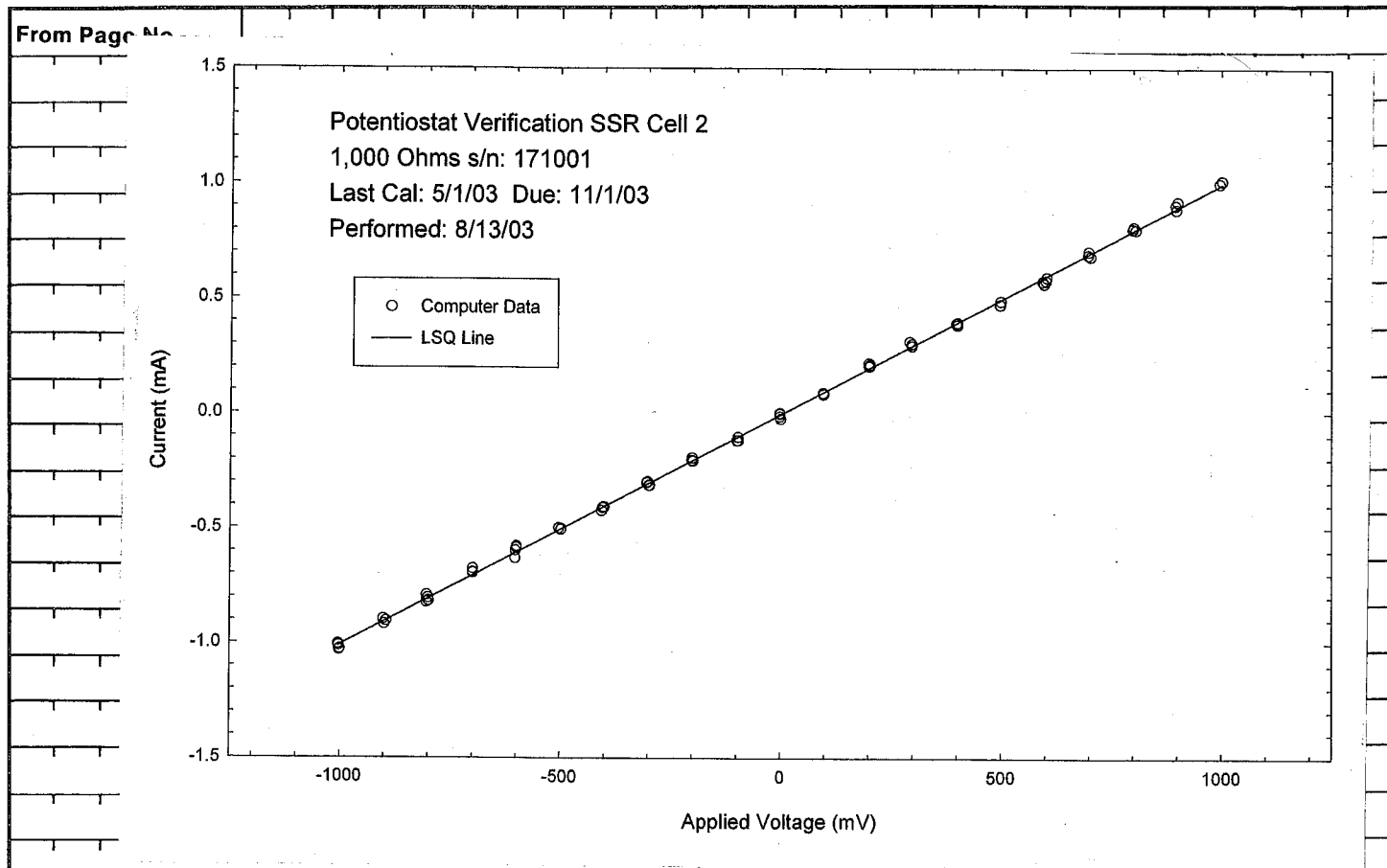
DI WATER TO MAKE 2L PH = 8.953

NEED 2.8L SOLUTION PER TEST

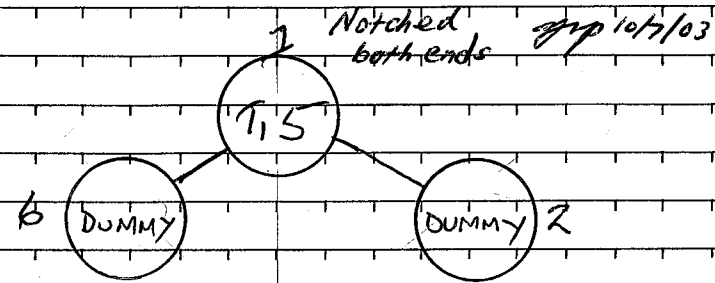
*AS*  
9/1/03 FINAL PH = 8.725

To Page No. \_\_\_\_\_

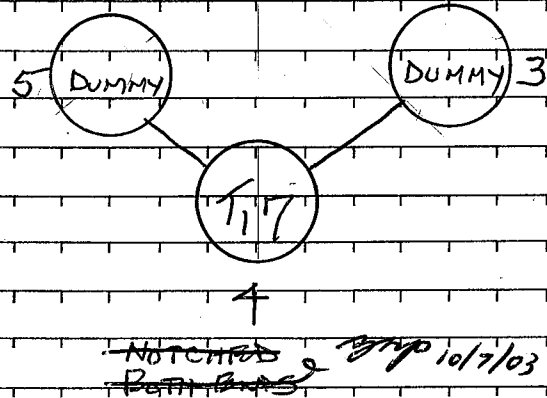
Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date *7/28/03*  
Recorded by *Jerry Schmit*



TEST SETUP



DUMMY IS LOW CARBON STEEL



To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

*Jerry Swartz*

8/15/03

From Page No. \_\_\_\_\_

JACK GEAR BOX

$\frac{1''}{100 \text{ TYPES}} \cdot \frac{1 \text{ TURN}}{1000 \text{ REV}} \cdot \frac{1 \text{ REV}}{1 \text{ SEC}} = \frac{1 \text{ INCHES}}{\text{SEC}} = 1E-7$

$\frac{1}{T} = 100 \cdot 1000 \cdot 1E-7 = 0.01$

$T = 100 \text{ SECONDS}$

T1 TEST 1 BKR 21/23

LOAD CELL 2 2MNA CL + 0.1MNA F

GRADE 5

FILES: T15GT1 T17GT1

8/25/03 0CV -575 mV

2:52 START DEAIRATING -720 mV

8/26/03 7:20 -725 mV

8:31 START HEATING SP 95°C -727 mV

9:15 37°C -730 mV

10:00 47°C -735 mV

10:30 57°C -740 mV

12:00 61°C -744 mV

1:00 64°C -760 mV

2:00 67°C -762 mV

2:45 68°C SPA 120°C -765 mV

3:45 76°C -730 mV

8/27/03 7:09 89°C SPA 126°C -732 mV

8:05 91°C -731 mV

9:05 91°C SPA 130°C

10:07 START LOG

10:10 START LOADING

8/28/03 7:00 COMPUTER PROBLEMS - STOP LOAD TRAIN -

LAST LOG ENTRY 8/28/03 4:17

NEW FILE NAMES: T15GT1A & T17GT1A

8/18 START LOADING

8/23 10:19 T17GT1A FAILED

8/23 9:57 T15GT1A HAS FAILED - SHUTDOWN

FINAL PH 8.725

SPECIMEN IN POSITION 1 WAS NOTCHED. 3/B POSITION 4 ERROR IN NOTES?

T17GT1 LAST ENTRY 8/28/03 16:17:13 29.8362 HRS

T15GT1A FIRST ENTRY 8/21/03 8:37:01 GAP 16.33 HRS

OFFSET 46.1662 HRS

JHS  
9/10/03

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

*Jerry Swartz*

8/15/03

From Page No. \_\_\_\_\_

Ti TEST 2

1M NaCl

LOAD CELL  
1/2

GRADE  
5  
7 NOTCHED

FILES: T15GT2  
T17GT2

TEST SOLUTION: 175.33 g NaCl - FISHER LOT 905502  
DIY WATER TO MAKE 3L  
INITIAL PH = 7.538

9/1/03 2:18 START DEBUBBING

9/10/03 9:07 83°C OCV = 2705 mV  
9:38 616 mV

12:35 ZERO TIME - START LOG FILES

12:36 START LOADING

9/18/03 LAST SPECIMEN FAILED

FINAL PH = 6.457

ALL OPEN CIRCUIT VOLTAGE MEASUREMENTS WERE MADE  
USING SCE S/N 0249091. THIS APPLIES TO BOTH  
TEST 1 & TEST 2.

Copies sent to QA records

*[Signature]* 9/10/03

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

9/19/03

From Page No. \_\_\_\_\_

I have reviewed this scientific notebook and find it in compliance with QAP-001. There is sufficient information regarding procedures used for conducting tests, acquiring and analyzing data so that another qualified individual could repeat the activity.

*[Signature]* 9/29/2004

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by