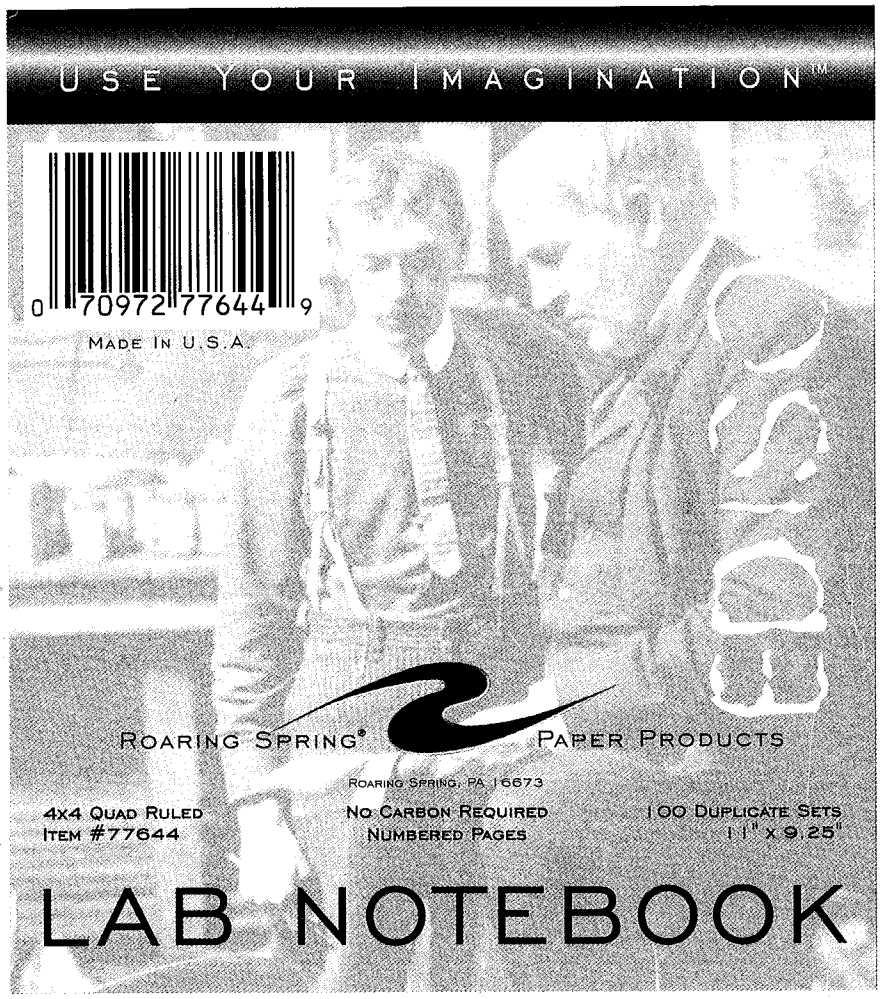

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COPY 893

"An experimental and analytical investigation of creep deformation behavior of titanium alloys" (P.O. # 713047)

The work involves experimental relating to microstructures and analysis of tensile and creep data of Grad 5 and Grade 7 titanium alloys.

The investigators have a Ph.D. in metallurgy or materials science

The entries in this lab note book are made by:

1. SREERAMAMURTHY ANKEM S. Ankem 7/16/07
2. PAUL G. OBERSON [signature] 7/16/07
3. Roman V. Kazban [signature] 9/14/07

Name: Dr. S. Amxem Date: 01/02/07 01

Experiment: Materials and data received to date.

A CD was received on 12/28/06 and it contained the following information: creep data for Grade 7 titanium Alloy at 150°C for nine different stress levels, 40, 55, 55, 70, 85, 100, 105, 110 and 115% of the yield stress. The identification of the specimens are:

D 58815, 100% Yield Stress
D 58516, 70% " "
D 58517, 85% " "
D 58518, 115% " "
D 58519, 55% " "
D 58520, 55% " "
D 58521, 40% " "
D 58523, 110% " "
D 58524, 105% " "

S. Amx

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 1/2/07

Experiment: Materials and Data Received (Cont)

2. FedEx Received on 12/30/06 containing 4 creep specimens: DS8521
DS8520
DS8516
DS8518

Greg Oberson

Name: Greg Oberson Date: 1/3/07

Experiment: Cutting Specimens for TEM

For undeformed specimens, I am cutting from the threaded ends of the specimen

Specimen 1: DS85/8

Equipment Used: Buehler Low Speed Saw with Diamond Wafering Blade

1/4/07

Experiment Continued

Punched 3mm diameter specimens from undeformed specimens to dimple

Equipment Used: South Bay Technologies Model 515 Dimpler

South Bay Technologies DCM060-S 6micron diamond paste

Specimens placed into TEM Grid Box I

Slots: A1, A2

1/5/07

Experiment Cont:

Slots A3, A4, A5, B1, B2

Name: Greg Oberson Date: 1/8/07 04

Experiment: Materials Received

From: Ken Chiang, SWRI
Via: Fedex

Creep Specimens, Ti Gr7, test temp = 150°C

Specimen ID	Stress Level
DS85S	100% YS
DS85S19	55% YS
DS85S23	110% YS
DS85S24	105% YS
DS85S09	Untested

Name: Greg Oberson Date: 1/8/07

Experiment: Jet Polishing TEM Specimens- Undeformed

Object: Prepare undeformed Gr 7 Ti specimens for TEM by using Jet Polishing

Equipment: Fischione 120 twin jet polishing unit

Polishing Solution: 120 mL methanol
4 mL H_2SO_4
4 mL HCl

Procedures:
1. Pour 120 mL methanol into large, round Pyrex dish in fume hood
2. Slowly add 4 mL H_2SO_4 to methanol
3. " " " " HCl " "
4. Pour ~~solution~~ methanol into rectangular Styrofoam container to depth of 0.75"
5. Place pyrex dish w/ polishing solution into methanol bath
6. Add dry ice to methanol bath until bubbling stops
7. Mount 3 mm TEM specimen into "Lollipop" holder
8. Place electropolish unit into Pyrex dish such that pumps are submerged
9. Connect wires to specimen holder
10. Turn 'Power' and 'Polish' switches on
11. turn 'Speed' dial to - 6
12. " 'Polish' " to - 10

Jet Polished 7 Specimens: A1 - B2

Name: Greg Dobson Date: 1/10/07

Experiment: Sample Prep for ESEM / Optical Microscopy

Cut threaded ends of specimen DS85S18 for polish/etch

polished face with SiC paper 240
400
600
800
1200 grit

then polish w/ cloth + colloidal silica for mirror finish

Name: Greg Oberson Date: 1/12/07

Experiment: Etching for Optical/ESEM

Etchants Used: Kroll's: 44 mL Water
5 mL nitric acid
1 mL hydrofluoric acid

Etch polished surface for ~ 1 min

Next: 'R' Etch: 16.5 g benzalkonium Chloride
40 mL glycerin
35 mL ethanol
25 mL HF

Etch for ~ 15 sec to Stain surface

GOM

Name: Greg Oberson Date: 1/16/07

Experiment: Ti Grade 7 Tensile Curves

Excel data from Ken.

2 specimens: DS85227 and DS85228

↓ YS = 32.9 ksi ↓ YS = 30.7 ksi

Elastic Strain: ~0.2%

See printouts on following pages.

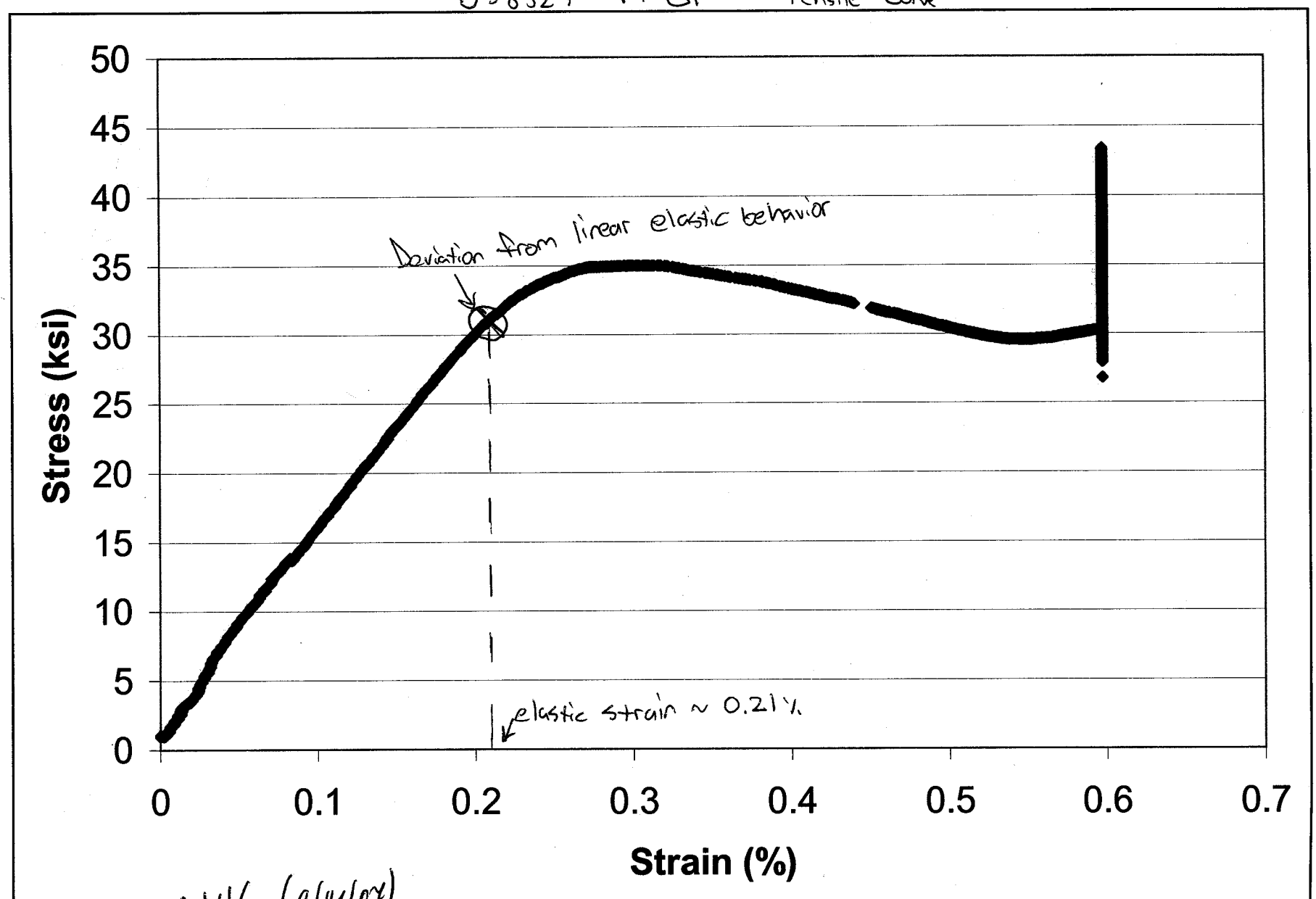
From as received creep curves, 0.2% elastic strain will be subtracted from given strain so that creep curves only reflect plastic strain.

Name:

Date:

Experiment:

D58527 Ti Gr 7 Tensile Curve



RVK (9/14/07)

Important: *Place card under blue copy.*

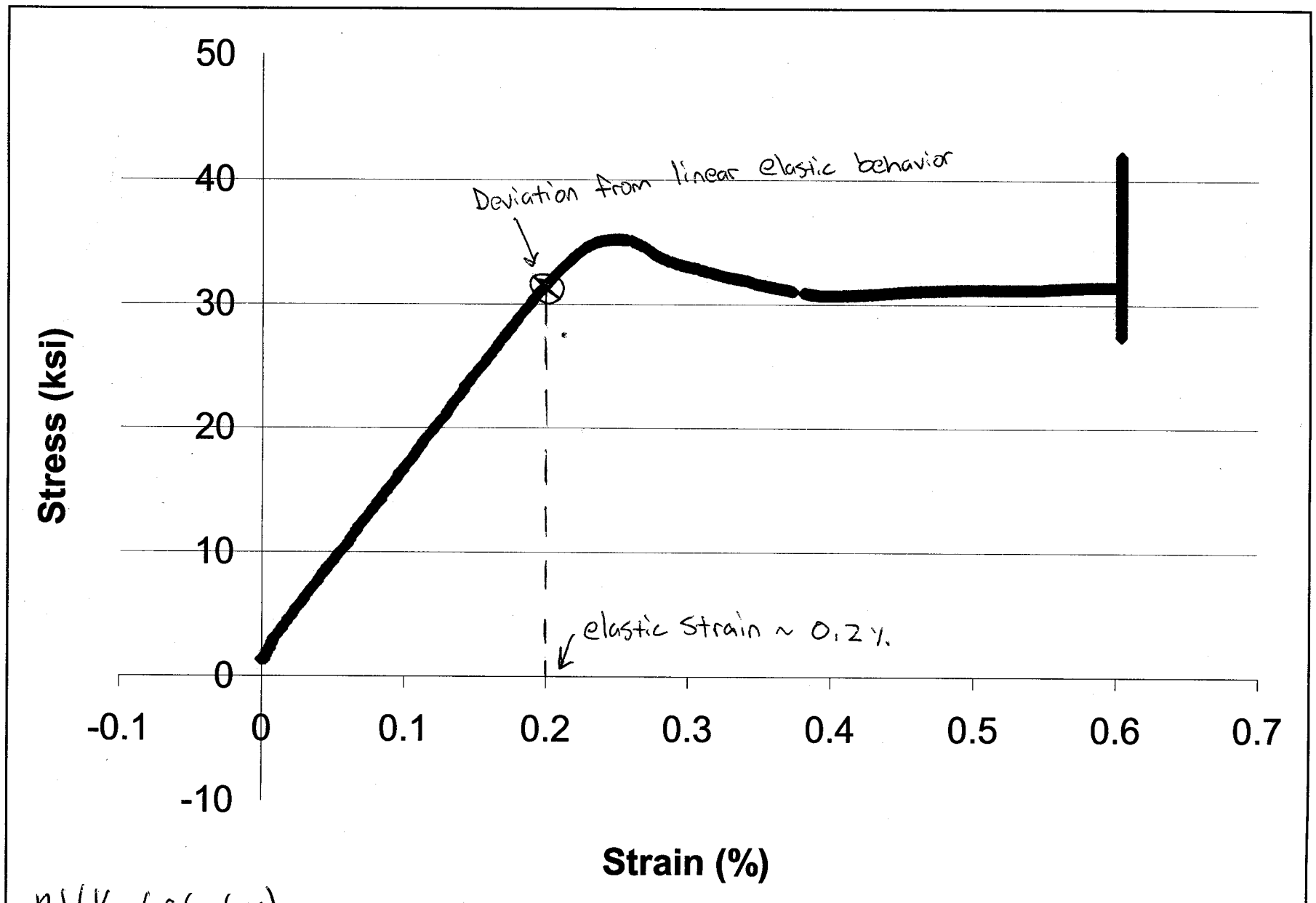
09

Name:

Date:

Experiment:

DS8528 Ti Gr 7 Tensile Curve



RVK (9/14/07)

Important: *Place card under blue copy.*

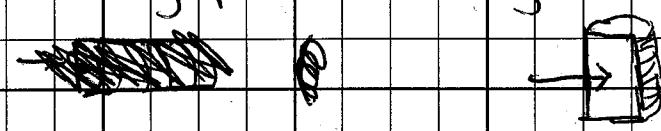
Name: Greg Oberson Date: 1/23/07

Experiment: Optical Microscopy for Undeformed Gr 7 Ti

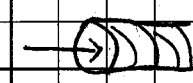
Objective: Measure Average Grain Size and Determine Morphology

Equipment: Zeiss ICM 405 inverted metallographic light microscope

Procedures: Micrographs taken ~~along~~ normal to tensile axis:

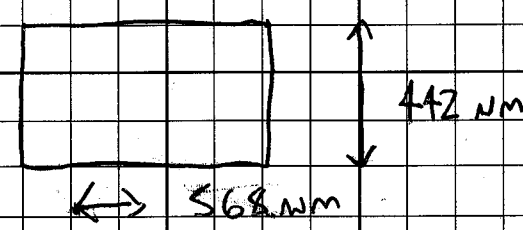


Micrographs taken along tensile Axis



To calculate average grain size, Follow ASTM Standard E112-96 using line intercept method (see attached)

Micrograph Dimensions



Pass	Grain Intercept Count	Orientation	d_{av}
1	11	H	56.8
2	14	H	40.6
3	15	H	43.7
4	10	V	49.1
5	11	V	44.2
6	14	H	40.6
7	12	H	51.6
8	13	H	47.3

Name: Greg Oberson Date: 1/23/07

Experiment: Optical Microscopy for Undeformed Grade 7 Ti

Pass	Grain Intercept count	Orientation	d_{av}
9	10	V	49.1
10	8	V	63.1
11	12	H	51.6
12	10	H	63.1
13	11	H	56.8
14	9	V	55.3
15	12	V	40.2
16	8	H	81.1
17	8	H	81.1
18	12	H	51.6
19	8	V	63.1
20	9	V	55.3
21	9	H	71.0
22	11	H	56.8
23	10	H	63.1
24	8	V	63.1
25	6	V	88.4
26	12	H	51.6
27	9	H	71.0
28	13	H	47.3
29	9	V	55.6
30	8	V	63.1
31	11	H	56.8
32	9	H	71.0
33	9	V	55.6

mean: d = 55.7 μm ± 6.3 μm

Name: Roman Kazlan Date: 9/14/04

Experiment:

Reference:

ASTM International. "Standard Test Methods for Determining Average Grain Size". E 112-96$^{\epsilon 3}$. ASTM International, 100 Barr Harbor Drive P.O. Box C700. West Conshohocken, PA 19428-2959, United States, p.p. 267-284

RK
9/14/04

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 1/23/07

Experiment: Cutting Deformed Grade 7 for TEM

- See procedures on p. 03
- Specimen Used: DS8S15 100% YS
- 8 Specimens (3 mm) disks cut from gage length
- Specimens will be dimpled following the procedure from p. 03
- Specimens placed into TEM Specimen Holder slots: B3, B4, B5, C1, C2, C3

Ron

1/25/07

- Cutting Specimens from DS8S16 : 70% YS
- Specimens placed into Specimen Holder slots: C4, C5, D1, D2, D3, D4

Ron

Name: Greg Oberson Date: 1/31/07

Experiment: Received FeDex from Ken Chiang

Specimens:

ES1196, Creep 1, Grade 7, 105% YS
ES1197, Creep 2, Grade 7, 110% YS } 302F
ES1198, Creep 3, Grade 7, 115% YS
ES1199, Creep 4, Grade 7, 85% YS, RT
ES1200, Creep 5, Grade 7, 85% YS, 50°C
ES1201, Creep 6, Grade 7, 85% YS, 100°C

DS 8517, Grade 7, 302F, 85% YS

Blocks of Gr 5 + Gr 7

Yon

Important: *Place card under blue copy.*

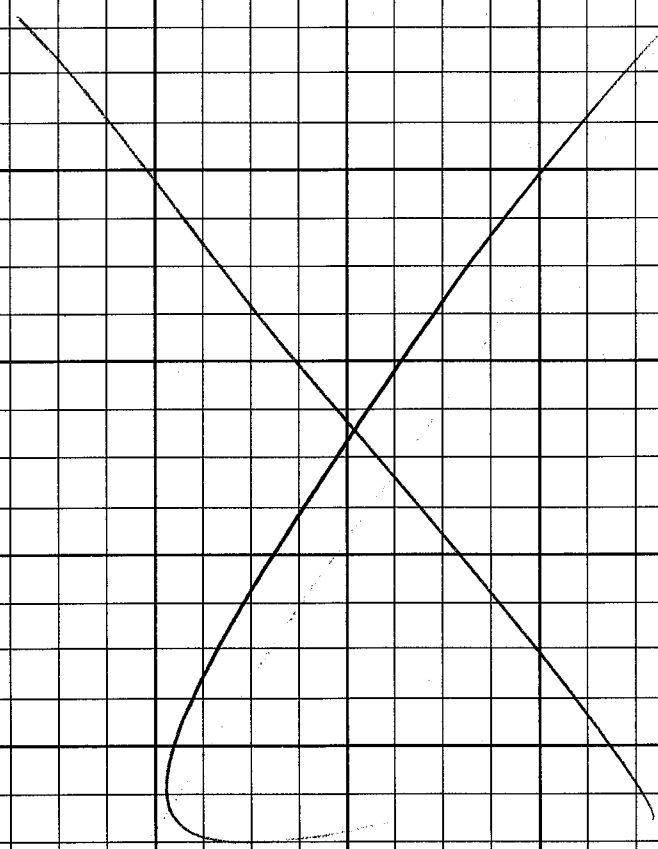
Name: Greg Oberson Date: 1/31/07

Experiment: Cutting Gr 07 specimens for TEM

See procedures on p. 03

Cutting Specimen D 588517, 302 F, 85% YS

Specimens in Slots D4 - E4



Name: Greg Oberson Date: 2/2/07

Experiment: Jet Polishing Grade 7 T: TEM Specimens

See procedures on pg. 05

Jet Polished 100% YS Specimens: B3-B5, C1-C3

Gon

Name: Greg Oberson Date: 2/15/07

Experiment: TEM

Specimen: G.07 100% YS: Specimen C in TEM holder

Used: JEM 2100 LaB_6 TEM

See images on disk

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 2/19/07

Experiment: Jet Polishing Grade 7 SS Y, YS 150°C Specimens

See Procedures on P. 05

In Specimen Holder Slots D4-E4

GO

Name: Greg Oberson Date: 2/27/2007

Experiment: TEM Ti Gr07, 85% TS, 150°C

Used: JeM 2100 LaB_6 Tem

Continued 3/1/07

See images on disk

Ger

Name: Greg Oberson Date: 3/7/07

Experiment: Cutting Gr 07 RT SSY, YS Specimens

Placed into specimen box in Slots: ES, A6-10

3/8/07

Jet Polished Specimens for TEM

3/9-3/10

TEM for above specimens
See images on disk

Name: Greg Oberson Date: 3/12/2007

Experiment: Cutting Gr 07 SSL YS 50 °C Specimens for TEM

Placed into specimen box in Slots: B6 - C6

3/13/07

Jet polished specimens, see procedures on p 63

3/14-15/07

Jem 2100 LaB_6 TEM investigation

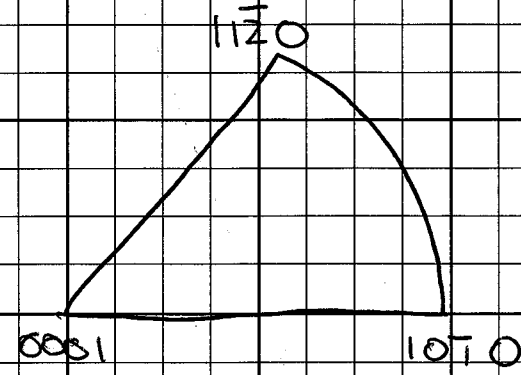
See images on disk

Name: Greg Oberson Date: 3/12/07

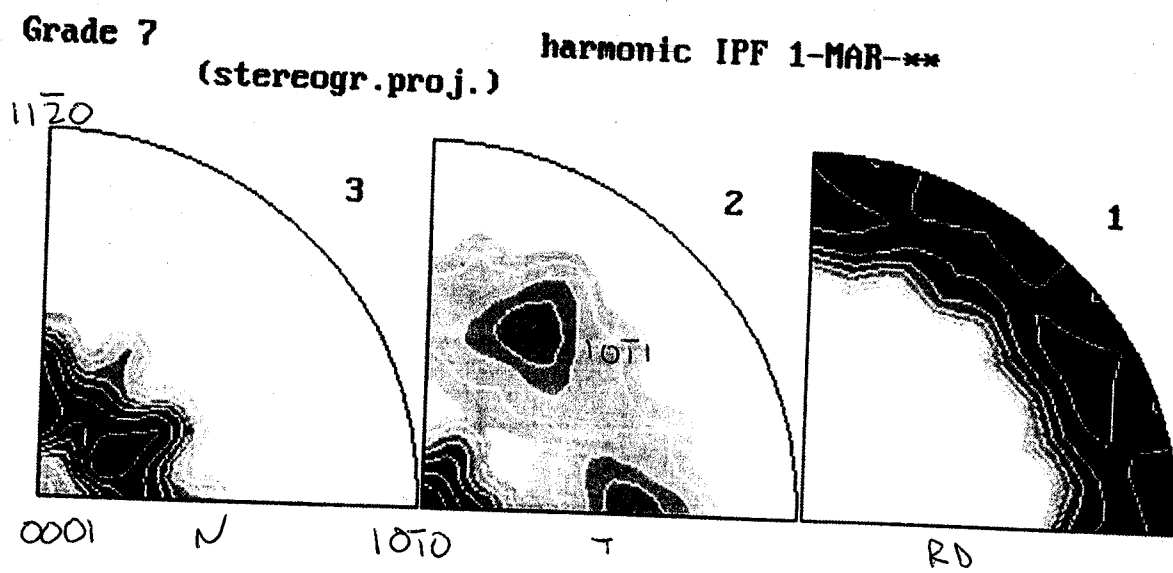
Experiment: Texture Analysis Gr 07 As Received

Pole figures received from Lambda Tech

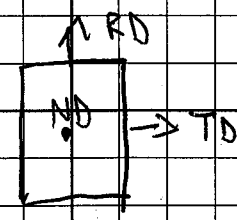
Orientation for inverse pole figures:

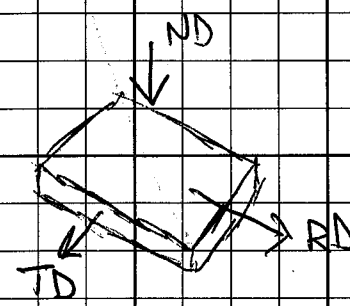


For reference see:
Jetter, et al
J. App. Phys., 27, 1956,
pp. 368-74



RVK





Important: *Place card under blue copy.*

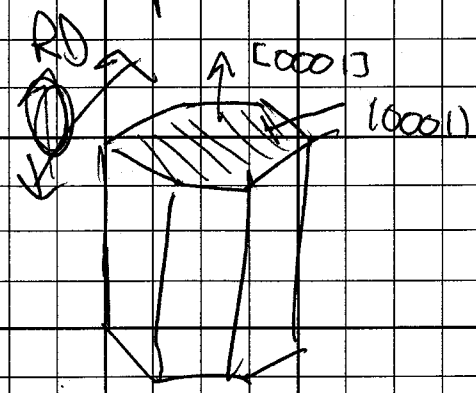
Name: Date:

Experiment:

Pole figures indicate:

Normal Dir'n: <0001> texture
Rolling Dir'n: <10$\bar{1}$0> texture
Transverse Dir'n: <10$\bar{1}$1> texture

graphically: Basal planes parallel to rolling plane



<10$\bar{1}$0> direction parallel to rolling direction

Name: Greg Oberson Date: 3/21/07

Experiment: Activation Energy for Creep at 150°C

Plot $\ln \dot{\varepsilon}$ vs $1/T$ at various strain levels

at RT:
0.2%, $\ln \dot{\varepsilon} = -10.07$
0.5%, $\ln \dot{\varepsilon} = -10.47$
1.0%, $\ln \dot{\varepsilon} = -11.09$
1.5%, $\ln \dot{\varepsilon} = -12.11$
2%, $\ln \dot{\varepsilon} = -12.95$
2.5%, $\ln \dot{\varepsilon} = -13.73$

at 50°C
0.2%, $\ln \dot{\varepsilon} = -9.25$
0.5%, $\ln \dot{\varepsilon} = -9.55$
1.0%, $\ln \dot{\varepsilon} = -10.19$
1.5%, $\ln \dot{\varepsilon} = -11.01$
2%, $\ln \dot{\varepsilon} = -12.01$
2.5%, $\ln \dot{\varepsilon} = -12.79$

at 100°C
0.2%, $\ln \dot{\varepsilon} = -8.03$
0.5%, $\ln \dot{\varepsilon} = -8.45$
1.0%, $\ln \dot{\varepsilon} = -9.17$
1.5%, $\ln \dot{\varepsilon} = -9.99$
2.0%, $\ln \dot{\varepsilon} = -10.99$
2.5%, $\ln \dot{\varepsilon} = -10.42$

at 150°C
0.2%, $\ln \dot{\varepsilon} = -7.96$
0.5%, $\ln \dot{\varepsilon} = -8.50$
1.0%, $\ln \dot{\varepsilon} = -9.93$
1.5%, $\ln \dot{\varepsilon} = -12.39$
2.0%, $\ln \dot{\varepsilon} = -14.56$

Name: Greg Dobson Date: 3/23/07

Experiment: Grain 100°C 85% YS Specimens for TEM

Placed into specimen Box, Slots C7-D1

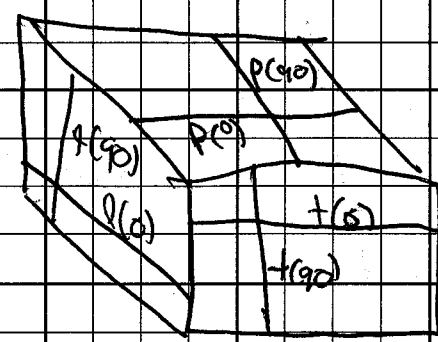
AC

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 3/26/07

Experiment: Calculating Avg Grain Size for Grade 5

Follow procedures from ASTM E112-96 for non-equiaxed grains:



$$\cancel{\bar{l} = \bar{l}(l(0))}$$

$$\bar{l} = \left[\bar{l}(l(0)) \cdot \bar{l}(t(90)) \cdot \bar{l}(p(90))\right]^{1/3}$$

$\bar{l}(0)$: longitudinal face, measured along RD

1 Micrograph: field of view: 121 μm
#int: 13
" : 10 → 11.7 μm
" : 11

2 Micrograph: field of view: 96 μm
#int: 9
#int: 7 → 16 μm
#int: 3

3 Micrograph: field of view: 52 μm
#int: 6
#int: 5 → 11.14 μm
#int: 6

Name: Date:

Experiment: Cont.

4 Micrograph: field of view: 136 nm
int: 11
int: 12 → 13.6 nm
int: 10

$l(0)$ = 13.1 nm

$t(90)$ Measurement: Along Short Face

Micrograph 1: Field of View: 61 nm
int: 13
: 14 → 4.9 nm
: 13

Micrograph 2: Field of View: 28 nm
int: 5
: 4 → 7 nm
: 6

Micrograph 3: Field of View: 58 nm
int: 12
: 12 → 5.1 nm
: 13

Micrograph 4: Field of View: 50 nm
int: 10
: 9 → 5.35 nm
: 12

$t(90)$ = 5.6 nm

Name: Date:

Experiment: Cont.

p(90) Measurement: Along planar face

Micrograph 1: Field of View: 256 μm
int: 23
int: 22 → 11.9 μm

Micrograph 2: Field of View: 140 μm
int: 7
int: 9 → 18.2 μm
int: 10

Micrograph 3: Field of View: 195 μm
int: 11
int: 8 → 20.17 μm
int: 13

p(90) = 16.58 μm

Avg Grain Size = (p(90) + t(90) + l(0))^1/3
= 10.7 μm

Name: Greg Oberson Date: 4/10/07

Experiment: Preparing Undeformed Gr OS for TEM

Specimens will be cut from the threaded end of the creep specimen

4/12/07

Specimens Dimpled, placed into Specimen Holder II, Slots: A1-B1

4/13/07

Jet Polish Specimens

Solution: 54% methanol - 110 mL
10% butanol - 13 mL
6% perchloric Acid - 8 mL

Voltage = 10 V

~ 30 min to polish through Specimen

4/16/07

TEM of Specimen A3

see digital images

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 4/18/07

Experiment: Cutting Deformed Grades for TEM

From specimen E6S402
150°C 85% YS

4/19/07

Specimens dimpled and placed into specimen holder in slots B2-C1

4/20/07

Specimens jet polished (see proc. on p. 30)

4/23/07

TEM 2100 LaB_6 of Specimen C3

See micrographs on disk

Don

Name: Greg Oberson Date: 4/25/07
Experiment: Cutting Grade 5 for TEM

Specimens cut from Specimen E6S403
150°C, 70%, 7S

Specimens dimpled and put into specimen
box in slots C2-D1

4/26/07

Specimens Jet Polished (see proc. on p. 30)

4/30/07

TEM of Specimen C4 JEOL 2100 LaB_6

See pictures on disk

Name: Greg Oberson Date: 5/1/07

Experiment: Cutting Grade 5 for TEM

Cut Specimens from E 653486

Room Temp, 485 ksi. YS

Specimens Dimpled and put into specimen box in slots D2-5

5/2/07

Specimens Jet Polished (See proc. on p.30)

5/3/07

TEM on JEOL 2100 LaB6 TEM

See images on disk

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 5/9/07

Experiment: Cutting Grade 5 for TEM

Specimens cut from E 655394
1000°C 85% YS

Specimens ~~[illegible]~~ Dimpled and put into Specimen Holder Slots E1-E4

5/10/07

Specimens yet polished, see procedures on p. 30.

5/11/07

TEM on JEOL 2100 LaB_6 TEM

See images on disk

Name: Greg Olsson Date: 5/14/07

Experiment: Cutting Grade 5 for TEM

Specimens Cut from E6S410, 250°C, 85% YS

Specimens Cut and put into specimen box

Slots ES-A9

5/15/07

Specimens Dimpled according to procedures on p 30

5/17/07

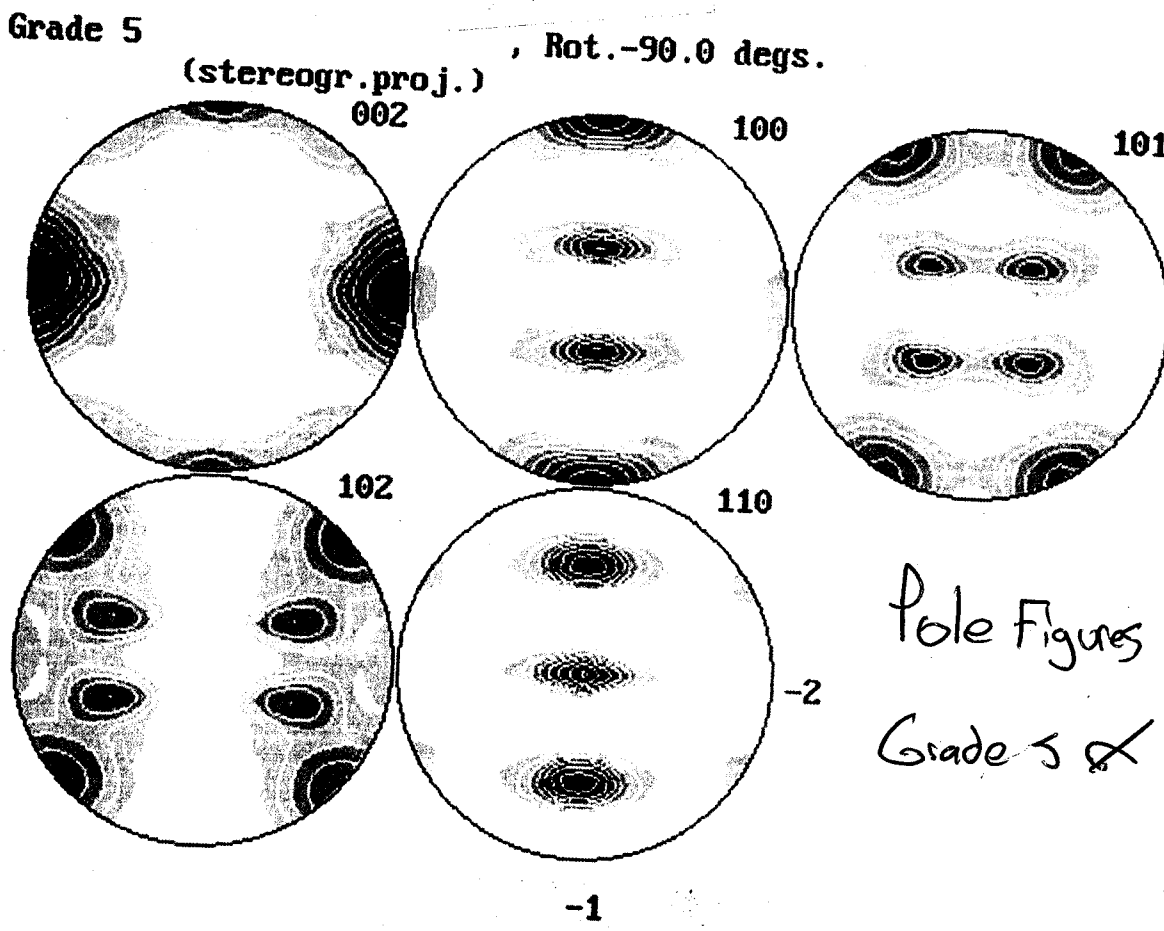
TEM on 2100 LaB_6

See images on Disk

Greg

Name: Greg Oberson Date: 6/1/06

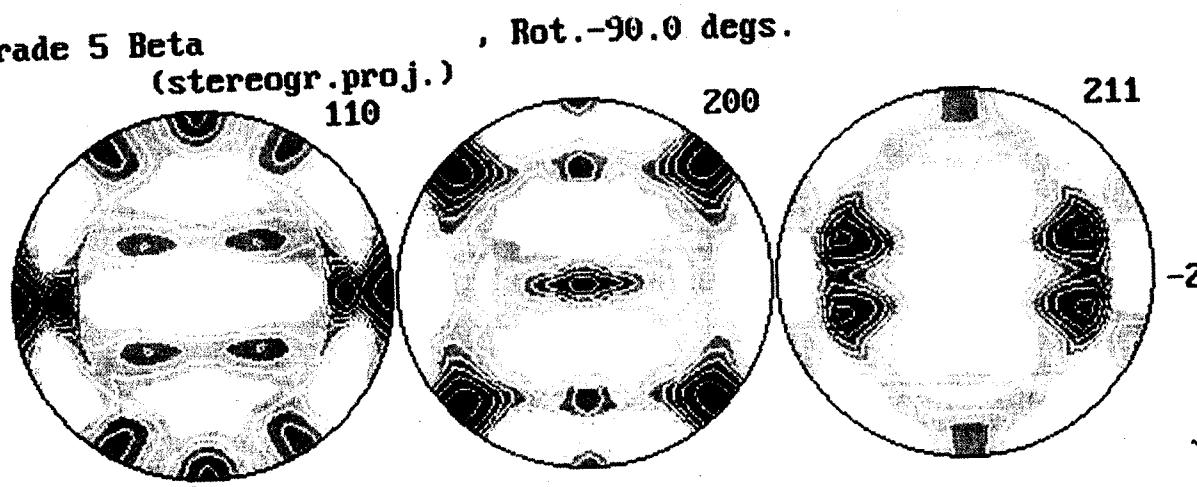
Experiment: Texture of Grade 5 Titanium



Pole Figures

Grade 5 α

RLK



Pole Figures Grade 5 β

RLK

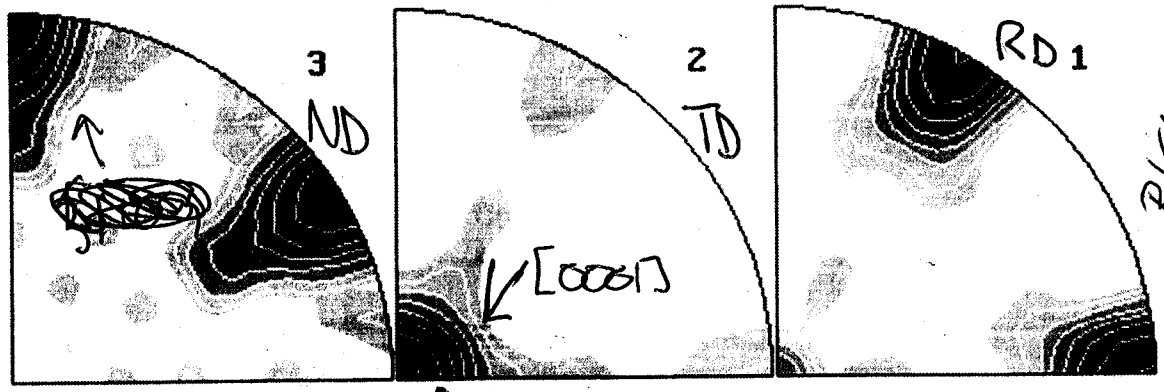
Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/1/06

Experiment: Texture Grade 5 Titanium

Grade 5 harmonic IPF 1-MAR-**

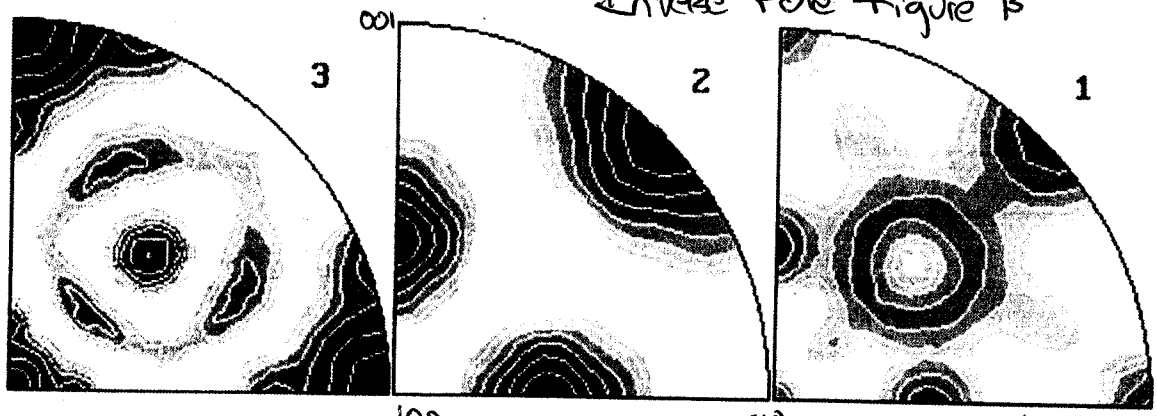
(stereogr.proj.) Inverse Pole figure α



RKK

Grade 5 Beta harmonic IPF 1-MAR-**

(stereogr.proj.) Inverse Pole figure β



RKK

{$\bar{1}$100} texture in RD α
{0001} texture in TD α
<11$\bar{2}$0> texture ND α
<110> texture TD β
<100> + <111> texture ND β

Name: Greg Olsson Date: 6/1/07

Experiment: Notes on Equipment Used

For Optical Microscopy:
Zeiss ICM 405 Inverted Light Microscope
Film: Polaroid Type 53 high-speed B&W film

For Scanning Electron Microscopy:
Electroscan Environmental SEM
operating @ 20 kV
Images taken on disk

For Transmission Electron Microscopy
JEOL 2100 LaB_6 TEM operating @ 200 keV
Images taken on film

Greg

Name: Greg Oberson Date: 6/1/06

Experiment: Procedures for Hand Polishing of as-Received Plate

Grade 5 and 7 as-received plates were hand-polished to reveal the microstructure.

The aim of polishing is to obtain a smooth and scratch free surface with no deformation products such as twins. The first step is to hand polish the surfaces of the specimen with silicon carbide grit paper. Relatively coarse 600 grit paper is used first followed by 800 then 1200 grit paper. With each successively finer grit, polish in a direction perpendicular to that of the previous grit until no scratches are visible to the naked eye in that direction. The specimen is rinsed with water and dried with compressed air every few minutes to reduce abrasion by residual particles. The 600 grit paper was used for approximately 10 minutes per side followed by 30 minutes per side for the 800 and 1200 grit. Buehler Mastermet® colloidal silica polishing suspension on a Chemomet® pad was used for the final polishing of the specimens. The specimen was polished for approximately 20 minutes on each side to achieve a mirror finish and until no scratches or surface features were visible under an optical microscope. The specimens were polished on three orthogonal faces.

RVR

Name: Greg Oberson Date: 6/1/07

Experiment: Procedures for Etching

Chemical etching is done in order to reveal the grain boundaries. Two different etchants are used. The first etchant is commonly known as Kroll's reagent, the composition of which is given in the table below. The specimen surface is etched very lightly with Kroll's reagent for approximately 20 seconds per side with a cotton swab. Kroll's reagent is a low-contrast etchant so the specimen is rinsed with water and etched with R-etch (composition given below) which stains the surface of the specimen. The surface is etched for approximately 10 seconds with R-etch.

RVK

Chemical Composition of Kroll's Reagent	
Chemical	Amount, mL [fl. oz.]
Hydrofluoric Acid - HF 50%	2 [0.068]
Nitric Acid - HNO_3	10 [0.34]
Distilled Water - H_2O	88 [2.98]

RVK

Composition of R-Etchant	
Chemical	Amount
Hydrofluoric Acid - HF 50%	25 mL [0.85 fl. oz.]
Benzalkonium Chloride	18.5 g [0.65 oz.]
Glycerine	40 mL [1.35 fl. oz.]

Name: Greg Oberson Date: 6/1/06

Experiment: Procedures for SEM

Operating Instructions for Electroscan Environmental Scanning Electron Microscope (ESEM)

1. Loading Specimen

1.1. Enter VACUUM menu and select VENT CHAMBER

1.2. After about 30 seconds, open chamber and remove stage.

1.3. Mount specimen on stage with grid lines facing up.

1.4. Replace the stage slowly and close the chamber door.

1.5. Enter VACUUM menu and select WET. The compressor requires about 90 seconds to create the vacuum in the chamber.

2. Obtaining an Image

2.1. When the message bar reads WET MODE, enter GUN menu.

2.2. Select HEAT FILAMENT. The filament requires about 4 minutes to stabilize.

2.3. While the filament is heating, go to the STAGE menu and select CALIBRATE. The stage will automatically position itself to the default position.

2.4. When the stage stops moving, turn on the chamber light.

2.5. Use the z-control switch next to the joystick to move the sample surface approximately 5 mm from the probe.

2.6. Push the SET button on the microscope and turn the water pressure dial clockwise until the readout is about 2.2. Push the SET button again.

2.7. Click the ON button for the voltage at the bottom left of the monitor.

2.8. Slowly increase the voltage to 20 kV with the arrows.

2.9. By looking into the chamber through the chamber window, use the joystick to position the grid surface directly below the probe.

2.10. Reduce the magnification below 100x and turn up the contrast until a white circle appears on the monitor.

2.11. Increase the magnification to over 500x. Press the P.F. button on the console.

Important: *Place card under blue copy.*

41b
(2 of 3)

2.12. Adjust the three silver knobs on top of the microscope by turning them one at a time and maximizing the height of the green line on the brightness monitor above the camera.

2.13. Press the P.F. button again to turn it off. Press the SOURCE button and decrease the magnification below 100x.

2.14. Open the left door below the console and adjust the left GUN BIAS know so the white circle on the monitor is as bright as possible.

2.15. Close the door. Press the SOURCE button again to turn it off and increase the magnification to over 500x.

2.16. Press the P.F. button on the console. Adjust the three silver knobs on top of the microscope by turning them one at a time and maximizing the brightness of the image.

2.17. Press the P.F. button again to turn it off. Press the ALIGN button.

2.18. Using the waveform on the oscilloscope as a guide, adjust the two aperture knobs on the chamber column so the green line is horizontal.

2.19. Press the ALIGN button again and turn it off. Press the P.F. button.

2.20. Adjust the three silver knobs on top of the microscope by turning them one at a time and maximizing the brightness of the image.

2.21. Press the P.F. button to turn it off.

2.22. Simultaneously increase the contrast and decrease the brightness until the brightness cannot be decreased.

2.23. Decrease the contrast so that the bottom of the green line touches the black horizontal line in the center of the oscilloscope.

2.24. Focus the image and adjust the X and Y stigmators properly to obtain a clear image.

3. Taking Pictures

3.1. Enter RECORD menu and select exposure time of 30 seconds.
3.2. Open Image Pro Plus software.

3.3. Click on icon of video camera (RECORD LIVE) to enable image capturing. Screen will show a live view of the magnified image.

Name: Greg Oberson Date: 6/1/00

Experiment: Procedures for SEM

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3.4. Press P.F. button and focus image. Press P.F. button again.

3.5. Press PHOTO button to obtain an image.

3.6. In Image Pro program, use sliders to obtain desired brightness and contrast.

3.7. Press SNAP to take picture.

3.8. Save image to ZIP disk as a TIF file.

4. Turning off Microscope

4.1. When microscopy is complete, enter GUN menu and select COOL FILAMENT.

4.2. Turn VOLTAGE to 0 kV and click the ON button to turn voltage off.

4.3. Enter VACUUM menu and click VENT CHAMBER.

4.4. Remove the sample from chamber and close chamber door.

4.5. Select WET from VACUUM menu.

4.6. When the WET mode is achieved, select STANDBY from the VACUUM menu.

4.7. Turn off chamber light.

4.8. Press SET button and turn water pressure to 0.0.

4.9. Press SET button again.

4.10. If last microscope user of the day, turn CONSOLE POWER off.

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/1/07 42a

Experiment: Procedures for TEM (1 of 4)

Operating Instructions for

JEOL 2100F Field Emission TEM

and

JEOL 2100 LaB_6 TEM

The following is a brief outline of the operating instructions for the JEOL 2100F Field Emission and LaB_6 TEM. This instrument should NOT be operated without proper training and certification by NISP Laboratory management. This outline is not a substitute for the official TEM operating manual.

PRELIMINARY STEPS

1. Load specimen onto double tilt holder. Specimen retainer is held down by a small screw. Make sure specimen is secure and centered. Check O-rings on the holder for lint which may compromise the vacuum.
2. Fill cold-trap to the left of the column with liquid nitrogen.
3. Raise High-Voltage Control window on the computer. Under Auto HT & Emission, click on StartUp to begin accelerating voltage. The status bar will show the time until the accelerating voltage of 200 kV is reached.

INSERTING SPECIMEN HOLDER INTO COLUMN

4. Open Specimen Property window on computer. Select Double Tilt Holder from drop down menu. Click Apply.

RVK

Important: *Place card under blue copy.*

42b
(2 of 4)

5. Align the specimen holder guide pin with the guide groove on the microscope column. Push the holder into the goniometer until it stops and set the goniometer PUMP/AIR switch to PUMP. The yellow light will turn on and the evacuation will begin. It will take about 10 minutes to pump down, until the green light on the goniometer goes on.
6. When the green light is on, turn the specimen holder clockwise and push the holder into the goniometer as far as it will go.
7. Turn holder fully clockwise and push in into the yellow light on the goniometer goes off.
8. Connect the cable from the end of the double tilt holder into the port above the specimen holder.

OBTAINING AN IMAGE

9. Press the BEAM VALVE button on the left side of the column to turn on the electron beam.
10. Press the LOW MAG button on the right side of the column and find the hole in the center of the specimen by using the sample translator trackball.
11. Turn BRIGHTNESS knob to increase or decrease the brightness of the beam and center beam using the SHIFT knobs.
12. Press STD FOCUS button to set the objective lens current to the reference value.
13. Microscope should now be aligned. The details of this procedure are too complex to include here. See operating manual for procedures.

RVK

Name: Greg Oberson Date: 6/1/07

Experiment: Procedures for TEM

42a (3 of 4)

RVR

TAKING A PICTURE

14. When ready to take a picture, press the PHOTO button on the right side of the column with the small screen placed in the electron beam path.
15. When the PHOTO button lights up, the film is at the photographing position. Press the PHOTO button again. The exposure time (when set to automatic exposure time) should be set using the small drop-down screen.

TURNING OFF THE MICROSCOPE

16. If specimen holder has been tilted, return tilt and azimuth to 0.
17. Decrease magnification to 5000x and press BEAM VALVE button to turn off the electron beam.
18. Go to Specimen Property window on the computer and click on Holder Exchange.
19. Specimen holder can now be removed from the column. Pull holder until it stops. Turn fully counterclockwise. Pull a little more. Turn fully counterclockwise again. Set PUMP/AIR switch to AIR and wait 30 seconds.
20. Remove holder from goniometer.
21. Raise High-Voltage Control window on the computer. Under Auto HT & Emission, click on Standby to decrease accelerating voltage.
22. Remove specimen from holder.

DEVELOPING MICROGRAPHS

23. Go to dark room and get empty film box and full film box from desiccator. Transport full film box in black bag and bring a second bag as well.

24. Turn off lights in microscope room. Open the door to the camera chamber by turning the handle clockwise until it stops. It will take about 5 minutes for the vacuum to be released and the door will pop open.
25. Slide magazine drawer out. Remove receiving magazine and place in film bag. Replace with an empty film box. Remove dispensing magazine and place in film bag. Replace with full film box.
26. Push magazine drawer back into chamber, close door, and turn handle counterclockwise.
27. Take film boxes back to dark room. Take negatives from receiving box one at a time and remove the negative from its frame. Place the negatives into the cradle being careful that they don't touch one another.
28. When all films are in cradle, place in developer and agitate. Developing should take about 1 minute or until numbers begin to appear at the scale bar. Be careful not to over or under develop the negatives.
29. After developing, rinse films in water for a couple minutes, then place cradle in fixer solution. The negatives must remain in the fixer for about 5-10 minutes.
30. While negatives are in fixer, refill the dispensing film box with new negatives. See the diagram in the dark-room for the correct way to place the pieces of film in the frames.
31. When dispensing film box is full, place it in the desiccator.
32. Remove negatives from fixer and rinse with water.
33. Place negatives in dryer for 30 minutes.

SCANNING NEGATIVES

34. Negatives are scanned on an Epson Perfection 4870 Pro scanner at a minimum resolution of 800 DPI. The files are saved as .TIF files.

RNR

Name: Greg Oberson Date: 6/1/07 43a

Experiment: Identifying Twin Modes in HCP Titanium (1 of 2

When using the TEM, twins are identified by obtaining selected area diffraction patterns (SADPs) of the matrix and the twin where the zone axis is the direction of the pole of rotation. The matrix and twin will have the same SADP but they will be rotated with respect to one another at an angle which is characteristic of the twinning mode. The most common twin in alpha-Titanium is the $\{1\bar{1}02\}\langle 10\bar{1}\bar{1}\rangle$ twin. In this case, the twin and the matrix are related by a rotation of approximately 85° along the shear plane normal, as schematically illustrated below.

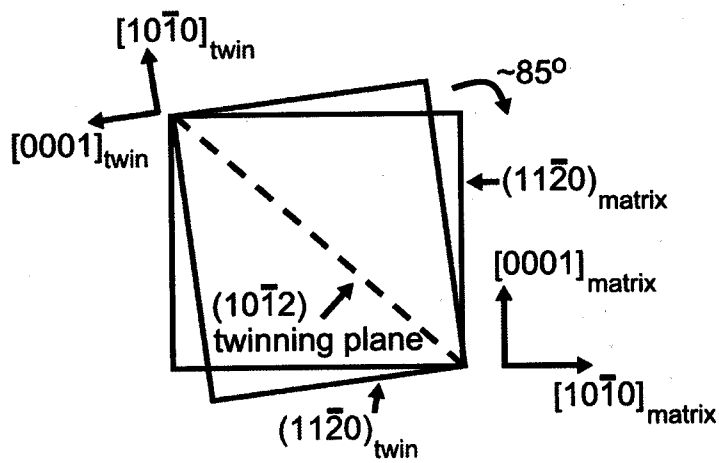


Figure 4.26: Schematic illustration of $\{11\bar{2}0\}$ planes of the matrix (black) and twin (red), respectively. In the case of the $\{1\bar{1}02\}$ twin, the $\{11\bar{2}0\}$ plane of the twin is rotated by a rotation of 85° (clockwise) with respect to the $\{11\bar{2}0\}$ plane of the matrix.

The less common twin is the $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ twin. In this case, the twin and matrix are related by a rotation of approximately 58° along the shear plane normal, as illustrated below.

Therefore, to identify the twinning mode, obtain a diffraction of the matrix where the zone axis is $\langle 11\bar{2}0\rangle$. Then, obtain a diffraction pattern of the twin with the same zone axis. Compare the angle of rotation between the diffraction pattern of the matrix and twin to identify the twin.

Name: Greg Obarson Date: 6/1/07

Experiment: Identifying Twin Modes in HCP Titanium

43b (2 of 2)

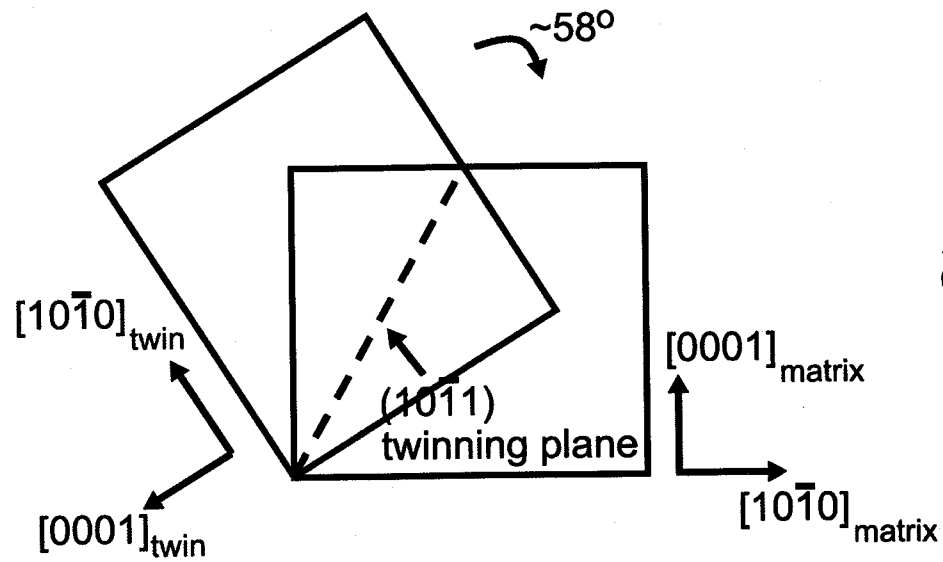


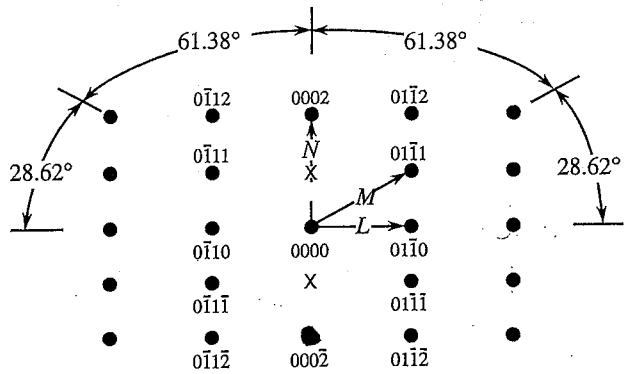
Figure 4.29: Schematic illustration of $\{11\bar{2}0\}$ planes of the matrix (black) and twin (red), respectively. In the case of the $\{1\bar{1}01\}$ twin, the $\{11\bar{2}0\}$ plane of the twin is rotated by a rotation of 58° (clockwise) with respect to the $\{11\bar{2}0\}$ plane of the matrix.

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/1/07

Experiment: Standard Diffraction Patterns in HCP α-Ti

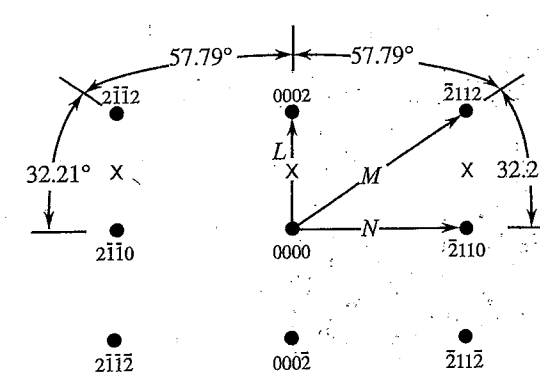
A



$\frac{N}{L}$ =1.09 $\frac{M}{L}$ =1.139 **B**=[2$\bar{1}\bar{1}$0]

foil plane (2$\bar{1}\bar{1}$0)

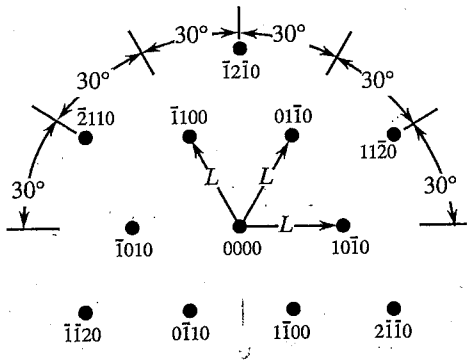
B



$\frac{N}{L}$ =1.587 $\frac{M}{L}$ =1.876 **B**=[01$\bar{1}$0]

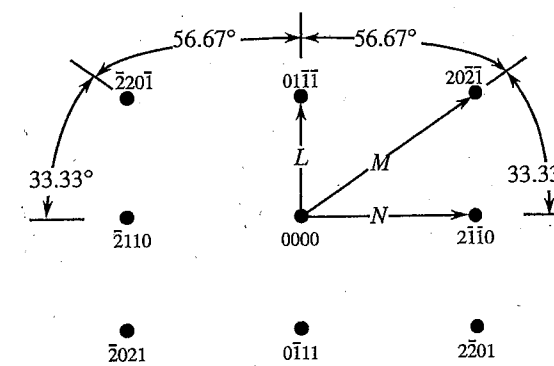
foil plane (01$\bar{1}$0)

C



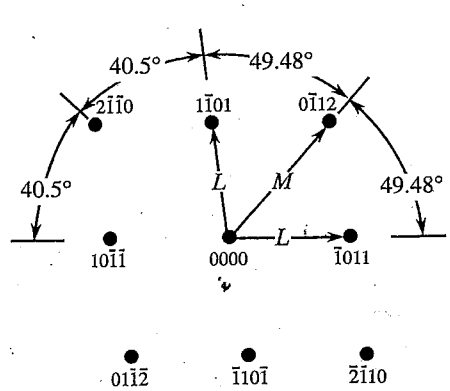
B=[0001] foil plane (0001)

D



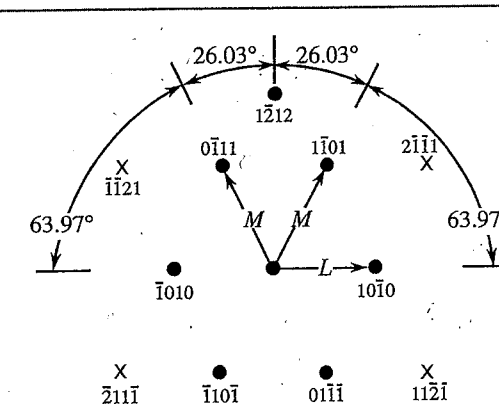
$\frac{N}{L}$ =1.520 $\frac{M}{L}$ =1.820 **B**=[01$\bar{1}$2]

E



$\frac{M}{L}$ =1.299 **B**=[01$\bar{1}$1]

F



$\frac{M}{L}$ =1.139 **B**=[$\bar{1}$2$\bar{1}$3]

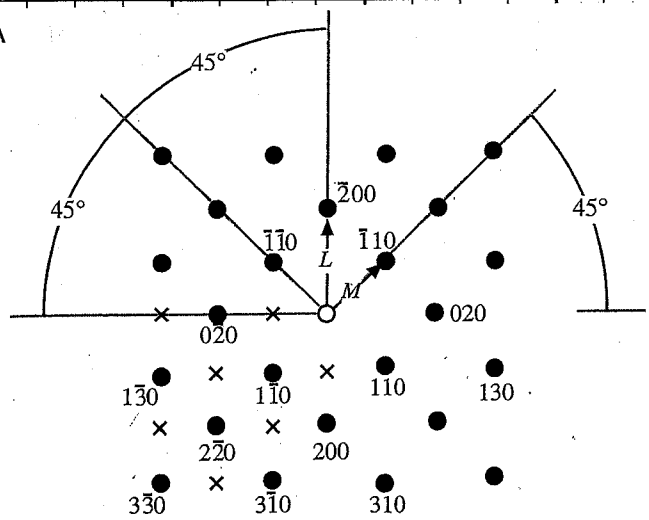
RVK

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/1/07

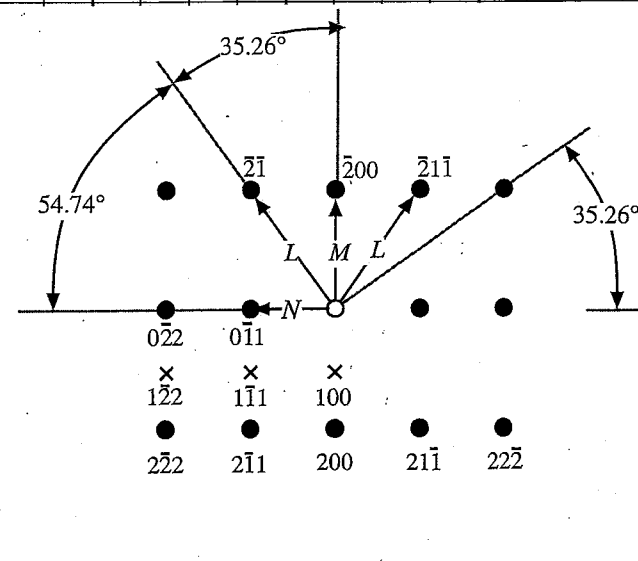
Experiment: Standard Diffraction Patterns of BCC β-Phase of Ø-BT:

A



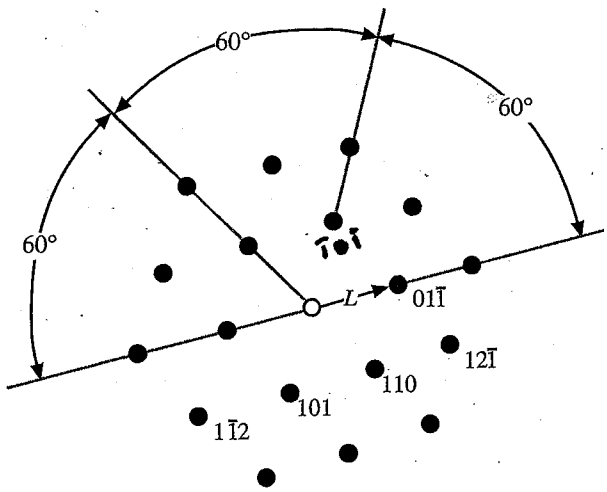
$\frac{L}{M} = \frac{\sqrt{4}}{\sqrt{2}} = 1.414$ **B** = [001]

B



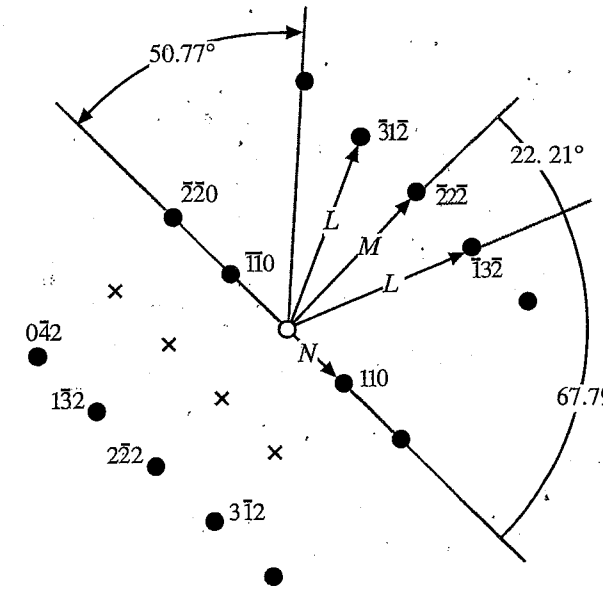
$\frac{L}{N} = \frac{\sqrt{6}}{\sqrt{2}} = 1.732$ $\frac{M}{N} = \frac{\sqrt{4}}{\sqrt{2}} = 1.414$ **B** = [011]

C



B = [$\bar{1}$11]

D



$\frac{L}{N} = \frac{\sqrt{14}}{\sqrt{2}} = 2.646$ $\frac{M}{N} = \frac{\sqrt{12}}{\sqrt{2}} = 2.450$ **B** = [$\bar{1}$12]

RLK

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/4/07

Experiment: Identifying Dislocations in Titanium

Attached is the procedure for identifying the Burgers vector, b, of a dislocation using g.b analysis. Taken from J.W. Edington, "Practical Electron Microscopy in Materials Science." N.V. Philips, 1976

9/14/07

Attachment was taken out. Please refer to J.W. Edington "Practical Electron Microscopy in Material's Science." N.V. Philips, 1976 pp. 60-61 and pp. 118 - 130

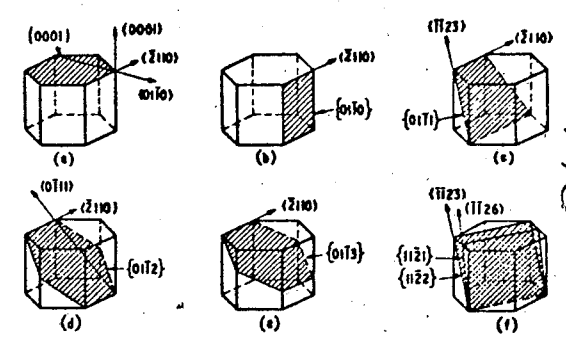
RNK

Name: Greg Oberson Date: 6/4/07

Experiment: Crystallography in Titanium

Table II. Indices of directions and planes

Directions		
Miller indices 3 axes	Miller–Bravais indices 4 axes	
[UVW]	4 index [uvtw]	3 index [uv.w]
* {001	0001	001
*{330	$11\bar{2}0$	110
030	$\bar{1}2\bar{1}0$	$\bar{1}20$
300	$2\bar{1}\bar{1}0$	$2\bar{1}0$
031	$\bar{1}2\bar{1}1$	$\bar{1}21$
012	$\bar{1}2\bar{1}6$	$\bar{1}26$
*{333	$11\bar{2}3$	113
033	$\bar{1}2\bar{1}3$	$\bar{1}23$
$\bar{3}03$	$\bar{2}113$	$\bar{2}13$
021	$\bar{2}4\bar{2}3$	$\bar{2}43$
*{210	$10\bar{1}0$	100
120	$01\bar{1}0$	010
$\bar{1}10$	$\bar{1}100$	$\bar{1}10$
211	$10\bar{1}1$	101
$12\bar{1}$	$01\bar{1}\bar{1}$	$01\bar{1}$
$\bar{1}11$	$\bar{1}101$	$\bar{1}11$
*{423	$20\bar{2}3$	203
243	$02\bar{2}3$	023
$\bar{2}23$	$\bar{2}203$	$\bar{2}23$
212	$10\bar{1}2$	102
122	$01\bar{1}2$	012
$1\bar{1}2$	$\bar{1}102$	$1\bar{1}2$
450	$12\bar{3}0$	120
310	$5\bar{1}\bar{4}0$	$5\bar{1}0$
$2\bar{1}0$	$5\bar{4}\bar{1}0$	$5\bar{4}0$
311	$5\bar{1}\bar{4}3$	$5\bar{1}3$
$2\bar{1}1$	$5\bar{4}\bar{1}3$	$5\bar{4}3$
411	$7\bar{2}\bar{5}3$	$7\bar{2}3$
*Indices of Burgers vectors	(hkil) Miller–Bravais indices	(HKL) Miller indices
	Planes	



8 Important planes and directions in a h.c.p. metal.

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/4/07

Experiment: Microstructures in Ti-6Al-4V

Taken from: Materials Properties Handbook: Titanium Alloys
R. Boyer and G. Welsch, eds, ASM Int'l 1994

Please refer to the reference pp. 488-489, pp. 608-617 for microstructures in Ti-6Al-4V

PVR 9/14/04

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/4/07

Experiment: Slip Systems in HCP α-Ti

Taken from: PG Partridge, Metall. Rev. Vol. 12, 1967, p 169

Please refer to the reference pp 178, 183–184 for slip systems in HCP α-Ti

RVR 9/14/07

Important: *Place card under blue copy.*

Name: Greg Oberson Date: 6/4/07

Experiment: Grade 7 Titanium Chemistry

50 a (1 of 3)

TRICOR
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(936) 273-2661
FAX (936) 273-2669
ho@tricormetals.com

INDUSTRIAL FASTENERS AND SUPPLIES

TRICOR METALS DIVISION

CERTIFICATE OF TESTS

CUSTOMER: SOUTHWEST RESEARCH INSTITUTE CUSTOMER PO#: 544447G

REPORT DATE: 10/28/03 SHIP DATE: 7/12/05 HEAT #: CN2775
LOT/TEST: 4-J5302-1-1 & 2-1

SIZE: .500"THK X 12" X 12"

FINISH: TITANIUM PLATE

MATERIAL SPECS. ASTM B: 265-98(A00), 01
ASME SB: 265-02 ALLOY: GRADE 7
EN10204 3.1.B

LBS/FT/PCS: 12.5 LBS ORDER #: 198825

CHEMICAL ANALYSIS %

C: .010/.010 H: 3/2/14 PPM O: .137/.139

N: .006/.007 Fe: .07/.08 Ti: BAL Pd: .15/.15

Hf: Fe+Cr: Zr+Hf: Ru:

MECHANICAL PROPERTIES

KSI-L	KSI-T
YIELD: 41/42	YIELD : 56/56
TENSILE: 72/74	TENSILE : 73/74
ELONGATION: 29/31	ELONGATION : 31/31
RED. OF AREA: 52/53	RED. OF AREA : 50/51

FLATTENING: HYDROSTATIC:
REVERSE FLATTEN: PNEUMATIC:
FLARE TEST: HARDNESS:
BEND TEST: EDDY CURRENT:
ANNEAL: ANNEALED 24MINS @1550F AC ULTRASONIC:

REMARKS:

THIS MATERIAL DID NOT COME IN CONTACT WITH MERCURY OR DEVICES CONTAINING MERCURY AT TRICOR METALS DIVISION. WE CERTIFY THAT THE CHEMICAL ANALYSIS AND PHYSICAL TEST RESULTS APPLYING ON THE ABOVE ORDER NUMBER ARE CORRECT AND TRUE TO THE BEST OF OUR KNOWLEDGE AND BELIEF.

AUTHORIZED SIGNATURE. [signature]

RVK

Important: *Place card under blue copy.*

(2 of 3) 909

Project No._____
Book No._____ TITLE_____

From Page No.____

Information potentially subject to copyright protection was redacted from this location. The redacted material is an approved certificate from TIMET in 2005 for a chemical and mechanical analysis.

To Page No.____

Witnessed & Understood by me,	Date	Invented by	Date
	7/20/05	Recorded by He	

Notebook 729

RVK

TITLE_____ Project No._____
Book No._____

From Page No.____

Procurement plan for Titanium Grade-7

CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES QUALITY ASSURANCE PROCEDURE	Proc. QAP-016 Revision 8 Chg 0 Page 8 of 9

ATTACHMENT 1

CNWRA PROCUREMENT PLAN

The following information is required when purchasing quality affecting items, services, or materials from a supplier not listed on the SwRI Approved Suppliers List.

Item or Activity to Be Procured	Action or Tests Required to Assure Quality
☐ Laboratory Analysis	☐ Provide blind samples with known compositions to the non-ASL supplier to provide confidence in reported results (Preferred approach) ☐ Provide duplicate samples to the non-ASL supplier to provide confidence in reported results ☐ Other (describe)
☒ Metals, Chemical Standards or other samples	☒ Perform confirmatory analysis with an ASL supplier in accordance with QAP-018
☐ Special Processes	☐ (describe)
☐ Other (explain)	☐ (describe)

Proposed Supplier: Tricor Metals

Reason for not using an ASL supplier or for not placing this supplier on the ASL:

☒ No ASL supplier is capable of providing the required items/services.
☐ Desired supplier does not have a quality program.
☐ Other:

Acceptance Criteria:
Perform confirmatory analysis with an ASL supplier

Procurement Plan Initiated By: Xihua He Date: 7/5/2005

Approved: [signature] Date: 7/5/2005
Director of CNWRA Quality Assurance

Results of Receipt Inspection: ☒ Accept ☐ Reject NCR Number:_____

Reference Documentation Location: Notebook No. 729, Pg # 67

Performed By: He Date: 8/25/05

CNWRA FORM QAP-18 (05/2004)

To Page No.____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by He	8/25/05

50 e (3 of 3)

Name: Greg Oberson Date: 6/4/07

Project No.
Book No. TITLE

From Page No.

Copy of Letter to Staveley Services for Confirmatory Analyses of Ti Grade-7 Heat # CN2775

Xihua He
Phone: (210) 522-5194
Fax: (210) 522-6081
E-mail: xhe@swri.org

Dear Connie Casillas,

I will send you the heat of Titanium Grade-7 (Heat # CN2775) submitted for chemical analyses. Per our previous phone conversation I need to have the specimen analyzed for the Ti, Pd, Fe, C, H, O, and N. According to the vendor, the material heats are within the chemical composition ranges specified in ASTM B265-02. The chemical composition of UNS R52400, specified in ASTM B265-02 is provided below.

C: 0.08 max
Fe: 0.30 max
H: 0.015 max
N: 0.03 max
O: 0.25 max
Pd: 0.12-0.25
Ti: balance

I have provided a copy of ASTM B265-02, a copy of the alloy composition provided by the vendor, and a copy of the mill test report.

The chemical analyses performed by Staveley Material Testing should provide the chemical composition of the alloy heat and should specify the heat number of the material. The analyses should conclusively determine if the heat submitted are within the specifications listed in ASTM B265-02. Any discrepancy between the chemical analyses performed by Staveley Material Testing and the specifications of ASTM B265-02 should be clearly identified.

Please call me if you have any questions. Thank you for your assistance.

Sincerely

Xihua He

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	8/25/05

Note book 729

TITLE Project No.
Book No.

From Page No.

staveleyservices MATERIALS TESTING

STAVELEY SERVICES – MATERIALS TESTING
10005 Freeman Avenue, Santa Fe Springs, California 90670
Tel: (562) 946-1721 Fax: (562) 944-8389 www.staveleymt.com

Customer: CNWRA
Material: CP Titanium Grade 7
Specification: ASTM B265, Rev: 02

PO/SO: PO TO FOLLOW
P/N:

Lab No: 406142 Date: 08/11/05
Heat Number: CN2775
Other: Chem Sample

CERTIFICATION OF TEST

CHEMISTRY ELEMENT		MINIMUM REQ'S	MAXIMUM REQ'S	ACTUAL	COMMENTS
N2	Nitrogen		0.03	0.01	✓
C	Carbon		0.08	0.01	✓
H2	Hydrogen		0.015	0.001	✓
Fe	Iron		0.30	0.08	✓
O2	Oxygen		0.25	0.13	✓
OE	Other Elements Each		0.1	0.1	✓ Less than
OET	Other Elements Total		0.4	0.4	✓ Less than
Ti	Titanium	BAL		BAL	✓
Pd	Palladium	0.12	0.25	0.16	✓

Conforms to requirements. Comment: METHOD: ICP, XRF, COMBUSTION

MTM PAGE: 1 of 1

Respectfully Submitted
Mario Garcia
Q.A. Supervisor

Accredited Nadcap Materials Testing Laboratory

STAVELEY SERVICES - MATERIALS TESTING

STAVELEY SERVICES submits this certification as the confidential property of our client, it shall not be reproduced except in full, without the written approval of STAVELEY SERVICES. The recording of false, fictitious or fraudulent statements or entries on this document may be punished as a felony under federal law.

The purchased titanium Grade-7 meets compositional specification requirements for UNS R52400 as specified per ASTM B265-05.

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	8/25/05

Name: Greg Obeson Date: 6/4/07

Experiment: Grade 5 Titanium Chemistry

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RNK

CERTIFICATE OF TESTS

TRICOR INDUSTRIAL, INC.

CUSTOMER	SOUTHWEST RESEARCH INST	ALLOY	TITANIUM GR 5
CUSTOMER P.O.#	764387G	HEAT#	R8453
TRICOR ORDER #	231206	DESCRIPTION	.500" THK PLATE
SHIP DATE	12/5/2006	MATERIAL SPEC.	AMS 4911 H;DMS 1592F MIL-T-9046 J; AM 2 AB 1
LBS/FT/PCS	36.7 LBS	FINISH	

CORPORATE OFFICE
3000 Old Airport Rd.
P.O. Box 752
Wooster, Ohio 44691
(800) 421-5141
(330) 264-3299
FAX (330) 262-6678

INDUSTRIAL BRANCH OPERATION
823 West Bowman Street
Wooster, Ohio 44691
(800) 421-5141
(330) 264-3299
FAX (330) 262-7311
sales@tricorindustrial.com

INDUSTRIAL BRANCH OPERATION
5169 Guernsey Street
Bellaire, Ohio 43906
(740) 676-5010
FAX (740) 676-2401

OHIO METALS DIVISION
3000 Old Airport Road
Wooster, Ohio 44691
(800) 421-5141
(330) 264-3299
FAX (330) 264-1181
sales@tricormetals.com

TEXAS METALS DIVISION
101 S. Trade Center Pkwy.
Conroe, Texas 77385
(888) 987-4267
(936) 273-2661
FAX (936) 273-2669
ho@tricormetals.com

CHEMICAL ANALYSIS %

C	0.015	Ti	BALANCE
H	67/75 (PPM)	Al	6.219
Fe	0.150	Y	
O	0.176	Pd	
N	0.010	OTHER	V: 4.000

MECHANICAL PROPERITES

UTI TENSILED	KSi: L: 138/138 T: 161/159	ELONG%	L: 14/14 T: 14/14
YIELD	KSi: L: 124/122 T: 147/146	RA%	L: 24/22 T: 31/33

REMARKS PRODUCT ANNEALED 1 HR. @ 1400 F. A.C. MICROSTRUCTURE/IMMERSION ULTRASONICALLY TESTED AND ACCEPTABLE..

THIS MATERIAL DID NOT COME IN CONTACT WITH MERCURY OR DEVICES CONTAINING MERCURY WHILE AT TRICOR METALS TEXAS DIVISION. WE CERTIFY THAT THE CHEMICAL ANALYSIS AND PHYSICAL TEST RESULTS APPLYING ON THE ABOVE ORDER NUMBER ARE CORRECT AND TRUE TO THE BEST OF OUR KNOWLEDGE AND BELIEF.

AUTHORIZED SIGNATURE Connie Leonard

DATE 12/5/06

INDUSTRIAL FASTENERS AND SUPPLIES

PREMIUM METALS FABRICATION AND MILL PRODUCTS

Important: *Place card under blue copy.*

GEOSCIENCES AND ENGINEERING DIVISION

SCIENTIFIC NOTEBOOK REVIEW CHECKLIST RECORD

Scientific Notebook No. 893 Project Numbers: 20.06002.01.342

Accomplished

- [x] 1. Initial entries per QAP-001
- [x] 2. Dating of entries
- [x] 3. Corrections (crossed out, one line through w/initials/date)
- [x] 4. No White out used
- [x] 5. Page number visible on copy or original notebook
- [x] 6. In process entries per QAP-001
- [x] 7. Figure information present
- [x] 8. Text readable
- [x] 9. Copyrighted material is identified
- [x] 10. Permanent ink or type only
- [x] 11. Signing of entries (not required on each page)
- [x] 12. Electronic media in the scientific notebook properly labeled
- [x] 13. NRC Supplementary Scientific Notebook Questions are addressed.
- [x] 14. The independent, two person verification required by AP-019, Section 5.2.1.2(b) is complete

Any discrepancies must be resolved before notebook closeout.

==========

I have reviewed this scientific notebook and find it in agreement with QAP-001.

Manager's Signature 9-27-2007 Date

Attach this completed form to the last page of the notebook.

Form QAP-01 (03/2007)