# LABORATORY NOTEBOOK

CNWRA/SwRI

**CONTROLLED COPY NO.** 617

**NOTEBOOK NO.** 617

**ISSUED TO** Kuang-Tsan Kenneth Chiang

K. T. Chiang

**ON** October 3 **20**03

**DEPARTMENT** 20

**RETURNED** April 14 **20**09

Project No. 06002.01.081
06002.01.321
06002.01.322

**SCIENTIFIC NOTEBOOK COMPANY**
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SCIENTIFIC NOTEBOOK CO.



TITLE Initial Scientific Notebook Entry

From Page No.

**Initial Scientific Notebook Entry for Dry-Air Oxidation, Humid Air Oxidation, Passive Film.**

**Title:** Isothermal oxidation tests, cyclic oxidation tests, dry-air oxidation with deposited salt, oxygen 16/oxygen 18 experiments, microstructure analysis.

**Test Performed by:** Kuang-Tsan Kenneth Chiang

**Objective:** Study the oxidation kinetics, oxide morphology, and mechanism of passive film formation in dry air, humid air and thermal cycling conditions. Study the effect of fabrication processes such as welding and post-weld heat treatment on passive film formation.

**Equipment:** High Temperature Microbalance, Lindberg Furnace, Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Analysis (EDX), X-ray Diffraction (XRD), Secondary Ion Mass Spectroscopy (SIMS), transmission Electron Microscopy (TEM).

**Materials:** Alloy C-22, Alloy 825

**Specimen Specifications:** ASTM G54-77 for determining total depth of attack for static oxidation testing.

**Measurement Parameters:** Temperature of oxidation treatment, weight change after oxidation and thermal cycling.

**Required Level of Accuracy:** Temperature ±3°C for oxidation studies, weight measurement ±.005 mg.

**Uncertainty and Source of Error:** Duplicated oxidation specimens will be tested for each oxidation conditions.

See pg #75 Also,

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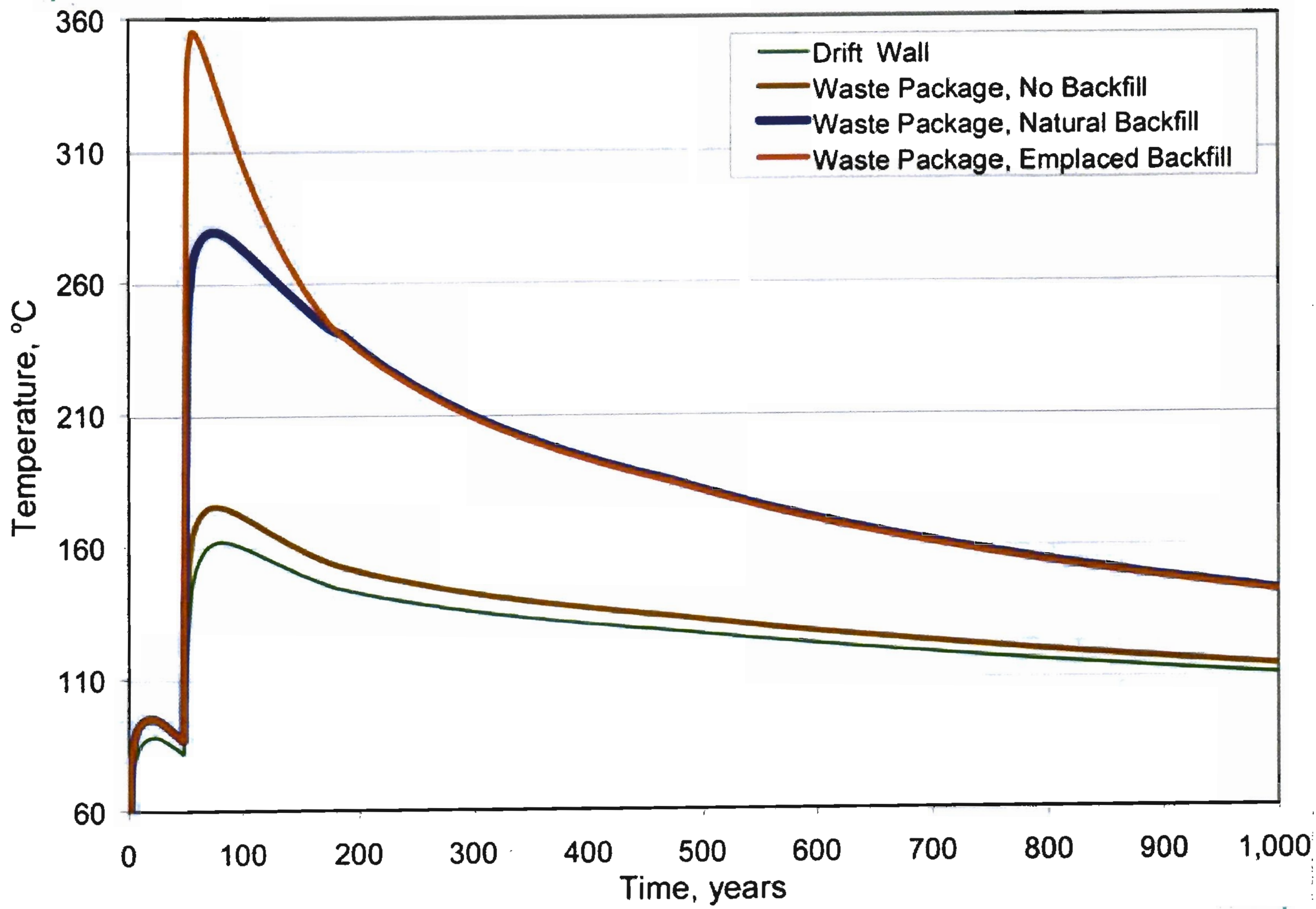
| | | K. T. Chiang | 10/8/03 |

Project No. 06002.01.081
Book No. 1 TITLE

From Page No. ___ Yucca Mountain Waste Package Surface Temperature Plot




**From:** Doug Gute [dgute@cnwra.swri.edu]
**Sent:** Tuesday, September 16, 2003 9:38 AM
**To:** Ken Chiang
**Subject:** Reference for the WP Surface Temperature Plot

Ken,

The reference for the WP Surface Temperature plot I sent to you is as follows:

Fedors R., G. Adams, C. Manepally, and S. Green. 2003. Thermal Conductivity, Edge Cooling, and Drift Degradation—Abstracted Model Sensitivity Analyses for Yucca Mountain. San Antonio, TX: Center for Nuclear Waste Regulatory Analyses.

Doug

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TITLE Schematic Diagram of Microbalance
Project No. 06002.01.081
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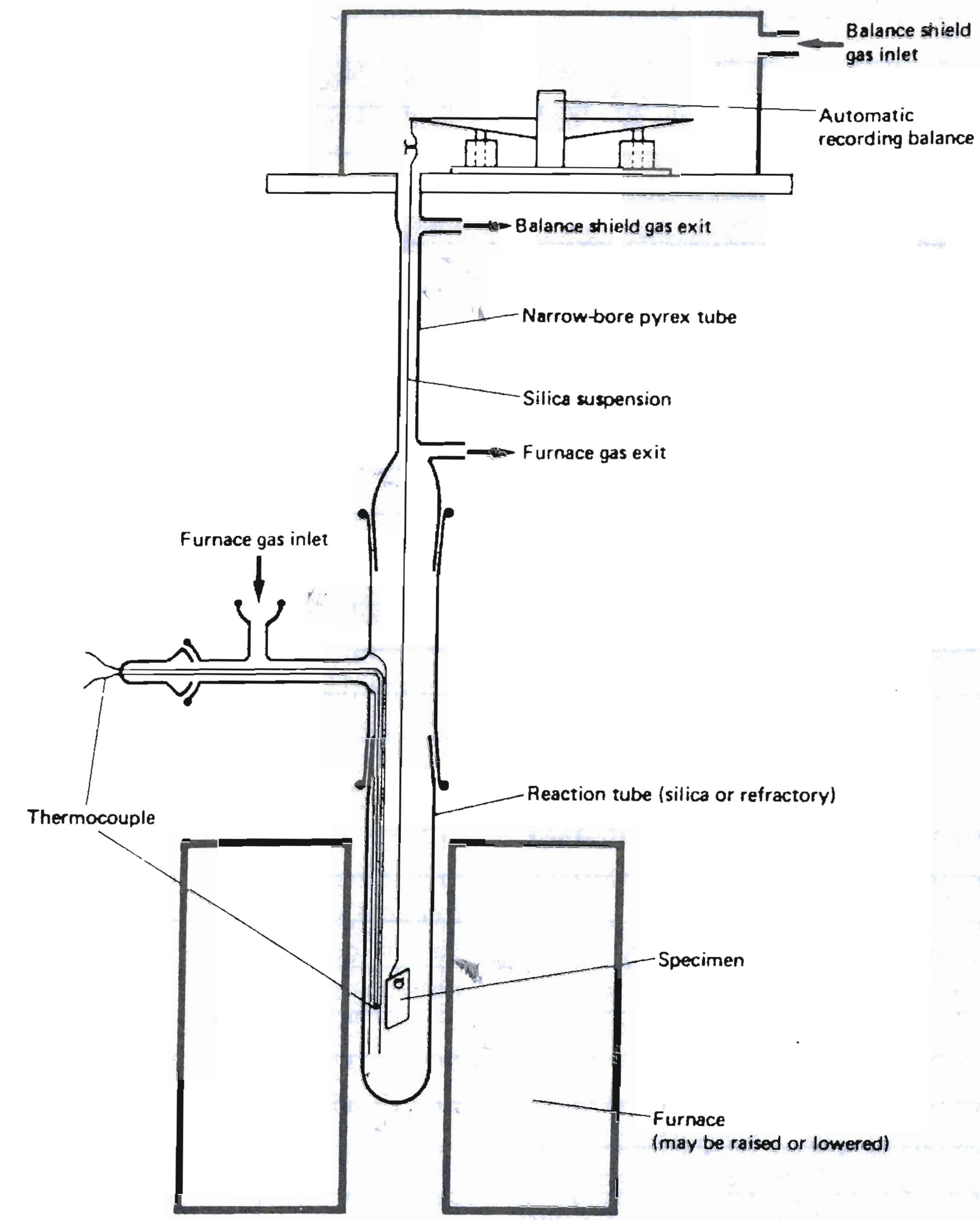
From Page No. ___ To measure kinetics of oxidation by monitoring the specimen weight change continuously.

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-----Original Message-----
**From:** Wes Patrick [mailto:wpatrick@cnwra.swri.edu]
**Sent:** Monday, August 25, 2003 7:22 AM
**To:** Dirs_Managers
**Cc:** Paul Maldonado
**Subject:** Capital Equipment Funding for FY2004

All,

Walt Downing has issued the subject request. Please analyze the capital equipment needs of your element or directorate (if not covered in the elements). Do so in the context of

(i) existing equipment,
(ii) outstanding requests for FY2003,
(iii) ongoing programs, and
(iv) business development needs.

I encourage you to engage your PIs with regard to items i-iii, and coordinate with David Ferrill with regard to item iv. In area iv, also consider situations where we may want to identify "contingency" capital equipment needs. This should be done where we plan to submit a proposal in an area where a capital item will be needed if the proposal is successful, but won't be needed otherwise.

I will need your input by close of business September 5th to allow time for internal evaluation before providing a consolidated request to Walt. At this time, we need to see the name of the equipment/item, a brief description of it, estimated cost, quarter when required, and a justification and any explanatory notes to support your request. Please note that purchase requisitions and capital equipment request forms are not needed at this time.

Thanks in advance.

Wes.

-----Original Message-----
**From:** Vijay Jain [mailto:vjain@cnwra.swri.edu]
**Sent:** Tuesday, September 02, 2003 8:56 AM
**To:** CSPE_Group
**Subject:** FW: Capital Equipment Funding for FY2004

Pleas eprovide your input this week.
Vijay



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| | | Recorded by K. T. Chiang | 10/20/03 |



**From:** Kuang-Tsan Ken Chiang [kchaing@swri.edu]
**Sent:** Friday, September 05, 2003 9:18 AM
**To:** 'vjain@swri.edu'
**Subject:** RE: Capital Equipment Funding for FY2004

The following is my input. We can discuss it this afternoon.
Ken

Name of the Equipment

D-100 Cahn Continuous-Recording Microbalance

Brief Description

Including weighing and control units, sample capacity of 100 grams, weighting range of ± 10 g with 10 micro gram maximum sensitivity, vacuum enclosure of weighing unit capable of 10-6 torr through separate vacuum port, Bake-out temperature of 125 °C, Gold anodized beam and teflon coated coil, RS-232 output, Automatic adjustment to electrical power between 100 and 240 volts, 50 to 60 Hz, weight for calibration, cables and Win NT/2000 based software for data collection and analysis.

Recommend ordering 002-003405-01 with it. This part is essential to mount the balance and make it easy to operate.
Estimated Cost

The D-100 costs $27,250.00.
Quarter When Required

First or second quarter.

Justification and Explanatory Notes to Support Request

In the service conditions for the canister in the in the YM repository, the container material Alloy 22 should begin service in dry oxidation followed by subsequent aqueous-dry corrosion conditions (cyclic wetting and drying). The proposed equipment will allow growth kinetics in dry oxidation to be studied. The results and the growth mechanism of the passive film will directly support the life prediction modeling of container materials.

**From:** Vijay Jain [vjain@cnwra.swri.edu]
**Sent:** Friday, September 05, 2003 2:50 PM
**To:** CSPE_Group
**Subject:** FW: Input on Capital Equipment Needs for FY 2004 due COB Today

FYI

-----Original Message-----
**From:** Wes Patrick [mailto:wpatrick@cnwra.swri.edu]
**Sent:** Friday, September 05, 2003 2:48 PM
**To:** Dirs_Managers
**Subject:** Input on Capital Equipment Needs for FY 2004 due COB Today

All,

Just a reminder. Let me know if you need another day or two, but the schedule is pretty short.

Wes.



Recorded by K. T. Chiang 10/20/03

**From:** Kuang-Tsan Ken Chiang [kchaing@swri.edu]
**Sent:** Wednesday, September 03, 2003 12:31 PM
**To:** CSPE_Group
**Subject:** Brain Storm Session - Friday 3-5 pm

We will have a brain storm session this Friday (9-5-03) 3:00-5:00pm after the Allegations Management training.
Location: Conference Room across from Vijay's office
Topic: What areas we can go out for external research funding?

Thanks in advance.

Ken

**From:** Kuang-Tsan Ken Chiang [kchaing@swri.edu]
**Sent:** Friday, September 05, 2003 5:28 PM
**To:** 'vjain@swri.edu'
**Subject:** RE: Input on Capital Equipment Needs for FY 2004 due COB Today

I have updated it following our team discussion.

Name of the Equipment

D-100 Cahn Continuous-Recording Microbalance

Brief Description

Including weighing and control units, sample capacity of 100 grams, weighting range of ± 10 g with 10 micro gram maximum sensitivity, vacuum enclosure of weighing unit capable of 10-6 torr through separate vacuum port, Bake-out temperature of 125 °C, Gold anodized beam and teflon coated coil, RS-232 output, Automatic adjustment to electrical power between 100 and 240 volts, 50 to 60 Hz, weight for calibration, cables and Win NT/2000 based software for data collection and analysis.

Recommend ordering 002-003405-01 with it. This part is essential to mount the balance and make it easy to operate.
Estimated Cost

The D-100 costs $27,250.00.
An upgaded version with 1700 degree C capability costs approximately $ 60,000.
Quarter When Required

First or second quarter.

Justification and Explanatory Notes to Support Request

In the service conditions for the canister in the in the YM repository, the container material Alloy 22 should begin service in dry oxidation followed by subsequent aqueous-dry corrosion conditions (cyclic wetting and drying). The proposed equipment will allow growth kinetics in dry oxidation to be studied. The oxide film growth kinetics, obeys either cubic, parabolic or some logrithmic rate law, will determine growth mechanism. The results and the mechanism of the passive film growth will directly support the life prediction modeling of container materials.

The upgraded version will expand high temperature corrosion capabilties to include studies of ceramic materials and thermal barrier coatings.

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From Page No. Requisition of a microbalance

Telephone conversation with Dr. Dun Chen, Thermo Cahn

**From:** Chen, Dun [dun.chen@thermo.com]
**Sent:** Wednesday, September 10, 2003 3:49 PM
**To:** kuang-tsan.chiang@swri.org
**Cc:** Jeff Eubanks (jeff.eubanks@thermo.com)
**Subject:** Data

Ken:

Nice to talk with you over the phone about your applications. As I mentioned to you, please contact Jeff about sending the samples, price, and such. At the least, here is a Word file which shows the run I made before with Chromel.

Please feel free to contact me, should you have any further questions.

Best Regards,

Dun Chen, Ph.D.
Senior Product Specialist
Thermo Cahn
Thermo Electron Corporation
Control Technologies Division
Material Characterization Business
5225 Verona Road
Madison, WI 53711
608-327-6779
608-358-9850 (cell)
608-273-6827 fax
Dun.Chen@Thermo.com
http://www.Thermo.com/Cahn

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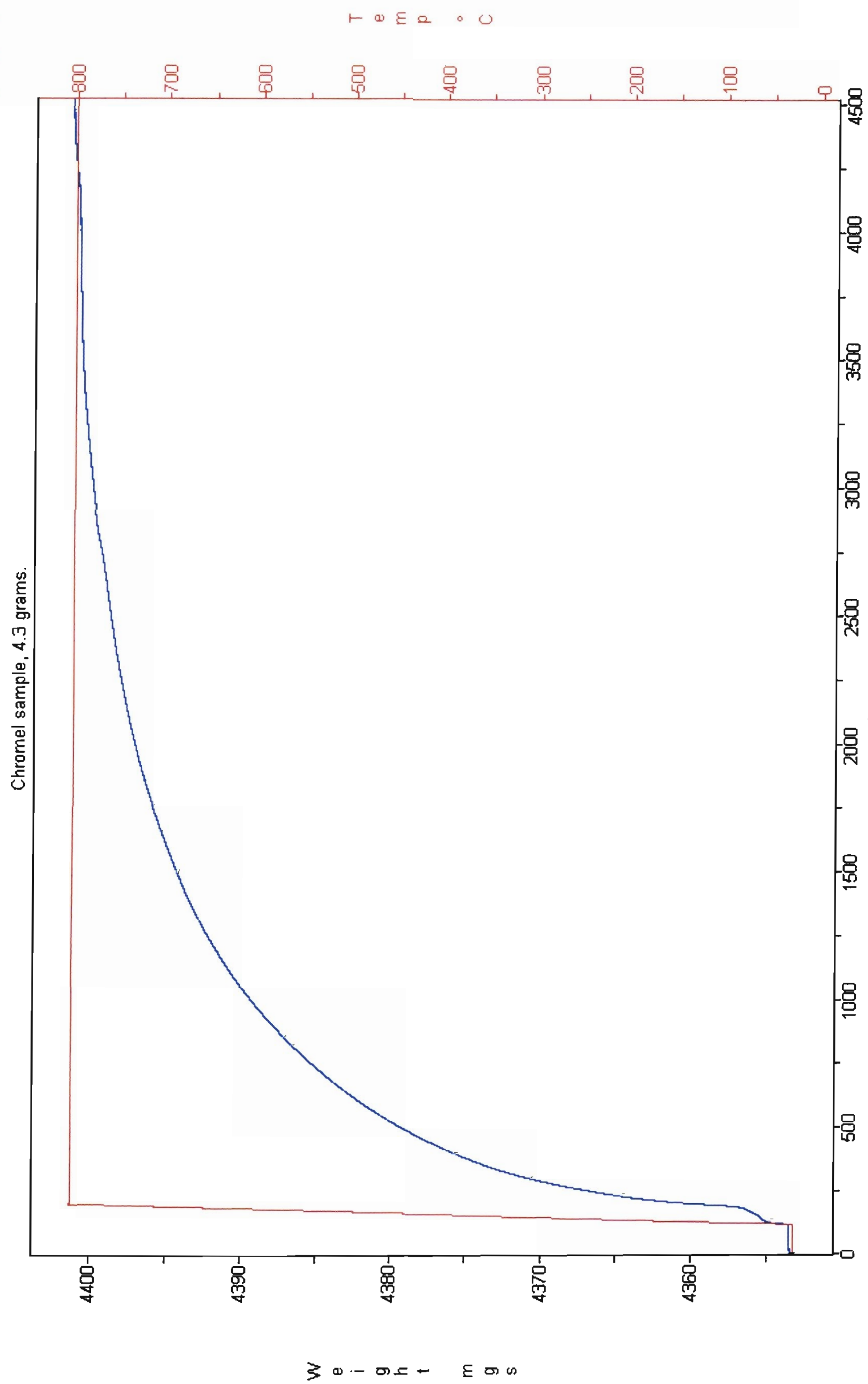
Example of TGA results

Red: temp.

Blue: wt change

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From Page No.

-----Original Message-----
**From:** Eubanks, Jeff [mailto:jeff.eubanks@thermo.com]
**Sent:** Monday, October 13, 2003 8:33 PM
**To:** kchiang@swri.edu
**Subject:** Thermo Cahn TGA

Ken,

Have you heard anything on your project being funded?

Are you still interested in our TGA products?

Did you ever decide which unit was best suited for your application and budget?

I would like to come by and introduce myself to you at your facility this week. Would this fit your schedule?

Jeff Eubanks
Sales Engineer
281-326-8030
jeff.eubanks@thermo.com
www.thermo.com/haake
Thermo Electron Corporation
Control Technologies Division
Material Characterization Business

-----Original Message-----
**From:** Kuang-Tsan Ken Chiang [mailto:kchiang@cnwra.swri.edu]
**Sent:** Wednesday, October 15, 2003 2:35 PM
**To:** 'Eubanks, Jeff'
**Subject:** RE: Thermo Cahn TGA

Jeff,

This week is bad time. Can we do it at a later date?

We are still interested in the TGA system. But we like to have some demonstration to determine the suitability of the units. Can we run some demonstration?

Ken Chiang
Senior Research Scientist
Southwest Research Institute
210-522-2308

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**From:** Eubanks, Jeff [mailto:jeff.eubanks@thermo.com]
**Sent:** Thursday, October 16, 2003 2:20 PM
**To:** kchiang@cnwra.swri.edu
**Subject:** RE: Thermo Cahn TGA

Ken,

The only option we have for demonstration of our TGA is at our lab in Madison, WI. We would very much like to offer you this demo. You could send us your samples ahead of time and we could provide you with the results before you arrive.

Please let me know how you would like to proceed.

Jeff Eubanks
Sales Engineer
281-326-8030
jeff.eubanks@thermo.com
www.thermo.com/haake
Thermo Electron Corporation
Control Technologies Division
Material Characterization Business

-----Original Message-----
**From:** Kuang-Tsan Ken Chiang [mailto:kchiang@cnwra.swri.edu]
**Sent:** Friday, October 17, 2003 5:12 PM
**To:** 'Eubanks, Jeff'
**Subject:** RE: Thermo Cahn TGA

Jeff,

I can prepare 4 specimens to be run in slow flowing air at 1100C, 850C, 600C, 350C. The 1100C and 850C samples will run for 24 hrs. The 600C and 350C samples will be run for 48 hrs.
The specimen dimension is approximately 13mmx19 mmx3 mm, weight 6.6 gm with a 1/16" hole drilled at center of one end for holding.

Can you perform this demo in the lab?

Ken Chiang
Senior Research Scientist
Southwest Research Institute

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From Page No.

**From:** Eubanks, Jeff [jeff.eubanks@thermo.com]
**Sent:** Monday, October 20, 2003 11:20 PM
**To:** kchiang@cnwra.swri.edu
**Subject:** RE: Thermo Cahn TGA

Ken,

I spoke to Dr. Dun Chen and he said that he could run your samples that you describe below. Do your samples degrade over time?

If not, see the file below which contains our application worksheet that needs to be filled out and sent in with your samples. The "ship to" address is contained in the form.

I would like to visit your facility this week, if your schedule allows, so that I can understand your expectations and your plans before we run your samples.

Let me know if this Thursday afternoon, October 23 would be a good time.

Jeff Eubanks
Sales Engineer
281-326-8030
jeff.eubanks@thermo.com
www.thermo.com/haake
Thermo Electron Corporation
Control Technologies Division
Material Characterization Business

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Material Alloy 22

From Page No.

**CNWRA SAMPLE CUSTODY LOG**
**PROC: TOP-012**

**Sample ID**: 2277-8-3175

**Log Notebook**: cnwra 009

**Purchase date**: March 14, 1989

**Log date**: April 11, 1994

**Entered by**: Sridhar

**Storage location**: Bldg 57

**Manufacturer**: Haynes International

**Processor**: Corrosion Materials

**Weight**:

**Length**: 44 in

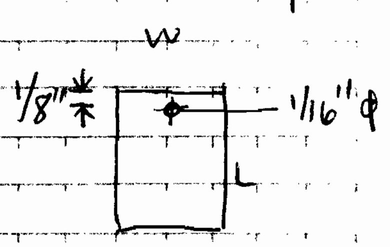
**Width**: 24 in

**Thickness**: 0.5 in

**Description**: Hastelloy alloy C-22 0.5 inch plate, Vendor analysis: 21.4 Cr, 3.8 Fe, 13.6 Mo, 3.0 W, 0.004 C (Primary melt analysis) 0.002 S, 0.008 P

4 C-22 specimens were prepared from machine shop

For each specimen, a 1/16" hole was drilled, center of width, the center of the hole was 1/8" from the edge.

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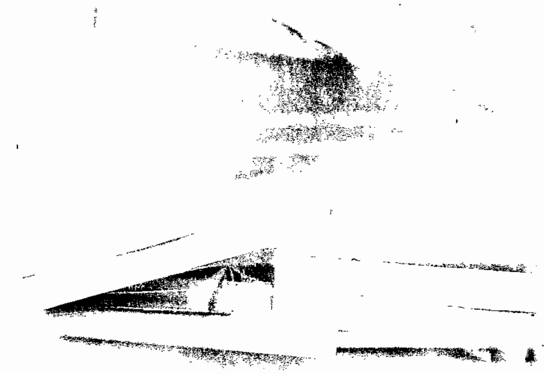
Initial Sample Dimension

specimens were polished through 600 grits SiC, dimension are:

C-22 Specimen
12.36 mm Width
3.04 mm Depth
19.37 mm Height
wt: 6.00689 g
Fisherbrand®

C-22 Specimen
13.37 mm Width
3.05 mm Depth
19.27 mm Height
wt: 6.57317 g
Fisherbrand®

Area = 4.788 + 0.751 + 1.178
= 6.717 cm²



C-22 Specimen
12.95 mm Width
3.04 mm Depth
19.32 mm Height
wt: 6.33033 g
Fisherbrand®

C-22 Specimen
12.57 mm Width
3.08 mm Depth
19.34 mm Height
wt: 6.19399 g
Fisherbrand®

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K.T. Chiang 11/17/03

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# Thermo

**Thermal Analysis & Surface Science**

Sample mail out 11/17/03

## Sample Request Form – Thermo Cahn Product Range

**Please send your samples to:**

Dr. Dun Chen
Thermo Cahn Instruments
5225 Verona Rd
Madison, WI 53711 – 4495
Phone: 608-327-6779
Fax: 608-273-6827

Salesperson: Jeff Eubanks
Phone: 281-326-8030

**MSDS sheets must accompany each sample**

Date: 11/17/2003

Customer Names: Ken Chiang, Ph.D.

Phone: 210-522-2308
Fax: 210-522-5184

Company: 6220 Culebra Road
Address: San Antonio, Texas 78238-5166

Results required by: Dec. 20, 2003

Technologies: TGA ☒ TG/FTIR &/or MS ☐
TGA/DTA ☐ DCA ☐
Other:

Sample Description: C22 Alloy specimens, Approximate dimension 13mm x 19mm x 3mm
Weight 6.6 gram
Please run TGA 1100°C - 24 hrs, 850°C - 24 hrs, 600°C - 48 hrs
350°C - 48 hrs in slow flowing air.

Expected Results: parabolic oxidation kinetics
Please return all tested specimens

Heating profile: (Include temperature range, heating rate, isotherm time, reaction environment & reaction gas flow rate where applicable).
maximum heating rate to temperature, keep constant temperature during exposure

Please attach any other details regarding the samples or anticipated results

Recorded by K. J. Chiang 11/17/03

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TITLE

From Page No. Shipping document

**SOUTHWEST RESEARCH INSTITUTE®**
6220 CULEBRA • SAN ANTONIO, TX 78238-5166
(210) 684-5111

4/17/0 Expl

SHIPPING TICKET NO. 422828 (THIS IS NOT A P.O. NO.)
DATE: 11/17/2003

**PRIOR TO REPAIR OF ANY ITEM MENTIONED BELOW, PLEASE CONTACT (210) 522-3074 WITH ESTIMATE AND FOR P.O. NO.**

SHIP TO:
COMPANY NAME: THERMO CAHN INSTRUMENTS
ATTN: DR. DUN CHEN
STREET ADDRESS: 5225 VERONA RD.
CITY/PROVINCE: MADISON, STATE/COUNTRY: WI ZIP CODE: 53711-4495
VENDOR PHONE NO.: (608)327-6779 FAX: (608)273-6827 R.M.A. NO.:

Please check box for method of shipment to and from Vendor
Circle specific method of shipment

| SHIP VIA | TO VENDOR | RETURN TO SWRI |
|---|---|---|
| MOTOR FREIGHT | ☐ | ☐ |
| AIR FREIGHT (PRIORITY AIR / GENERAL CARGO) | ☐ | ☐ |
| UPS (1 day, 2 day, 3 day or 7-10 days) | ☐ | ☐ |
| FedEx (1 day, 2 days) or FedEx Ground | ☐ | ☐ |
| SATURDAY DELIVERY | ☐ | ☐ |
| AIRBORNE (1 day, 2 day) | ☐ | ☐ |
| OTHER PRIORITY ONE | ☐ | ☐ |
| SWRI DRIVER | ☐ | ☐ |

PPD. ☒ COLL ☐ | INSURE FOR: | DECLARED VALUE: | GOVT. PROJ. ☐ | ORG. 20 | BLDG. NO. 57 | PURCHASE ORDER NO.:

AIR BILL OR W/B NO.: 6105 7535 5655 | ACCOUNT OR PROJECT NO.: 20.06002.01.081 | SWRI REQ. NO.: | SENDER'S NAME: KEN CHIANG | EXT. 2308

PERSON TO BE CONTACTED FOR PICKUP IS: | AT BLDG.: | EXT.:

| QUANTITY | DESCRIPTION & SERIAL NO. | ORIGINAL P.O. SOURCE C.O.D./REQ. NO. B/O |
|---|---|---|
| 1 BOX | C22 ALLOY SPECIMENS WT.6.6 GRAM APPROX. DIMENSION 13mm x 19mm x 3mm | |

Ref: 1.20 06002.01.081/422828 Date: 17NOV03 SHIPPING $8.22
Dept: DEPARTMENT 20 Wgt: 0.85 LBS SPECIAL $0.25
HANDLING $0.00
TOTAL $8.47
SERVICE: PRIORITY OVERNIGHT
TRACK: 6105 7535 5655

S.W.R.I. BUYER | EXT. | BUYER NOTIFIED ☐ | Please indicate if items are HAZARDOUS MATERIAL ☐ YES ☐ NO

NO. OF PACKAGES | TYPE OF PACKAGE ☐ BOX ☐ CRATE ☐ DRUM ☐ PALLET ☐ OTHER | WEIGHT | DIMENSIONS (Length x Width x Height)

**REASON FOR SHIPMENT AND/OR REMARKS**

☐ REPAIR (INDICATE PROBLEM IN REMARKS AREA) ☐ WARRANTY ☐ CREDIT ☐ CREDIT LESS RESTOCKING CHG. ☐ EXCHANGE ☐ OTHER (EXPLAIN IN REMARKS AREA)

REMARKS

DATE SHIPPED | VENDOR RECEIPT BY: | Dept. Approval Signature

THIS FORM TO BE USED FOR ANYTHING SHIPPED FROM S.W.R.I.

PACKING LIST (PINK COPY) SHOULD BE ATTACHED TO THE OUTSIDE OF THE CONTAINER, REMAINDER OF SHIPPING TICKET SHOULD BE SENT TO SHIPPING. PART 5 (GOLDEN COPY) WILL BE SENT TO YOU FOR DEPARTMENT RECORDS.

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Recorded by K. J. Chiang | 11/17/03

From Pa

Thermo Cahn

# EXPERIMENTAL REPORT

## Customer: Southwest Research Institute

Date: December 8, 2003

### Experimental Objectives

Four metal pieces were received from the customer. Their long term oxidation properties (over 1 or 2 days) at various isothermal temperatures were of interest.

### Experimental Set-up

Experiments were performed on a Thermo Cahn TherMax 300 system (replaced by VersaTherm HM). Sample pieces had a hole on them, and were hanged onto the system directly. Per customer's request, each one was subjected to a different isothermal temperature. Those were 1100 °C for 24 hours, 850 °C for 24 hours, 600 °C for 48 hours or 350 °C for 48 hours. To get there, a heating rate of 100 °C/min was used. Air was used as the reaction gas throughout the run to study the oxidation properties. Due to the customized sample shapes and weight, blank runs were not collected, and original data were presented here.

### Results

Figures 1 to 4 show the TG plots for the runs made. For runs at 600 °C and 350 °C, due to the very little weight change, zoomed in plots were showed as well to show detailed information about the data.

It can be seen from the figures that sample showed much larger weight gain at 1100 °C and 850 °C. However, much lower weight gains were seen at 600 °C (about 80 μg) and at 350 °C (about 25 μg), over 48 hours.

### Conclusions

Results showed the Cahn TG systems can be used to obtain the LONG term weight changes of the samples very easily and accurately. Better results can be obtained with better controlled lab conditions, and performing blank runs.

Dun Chen, Ph.D.
Senior Product Specialist.

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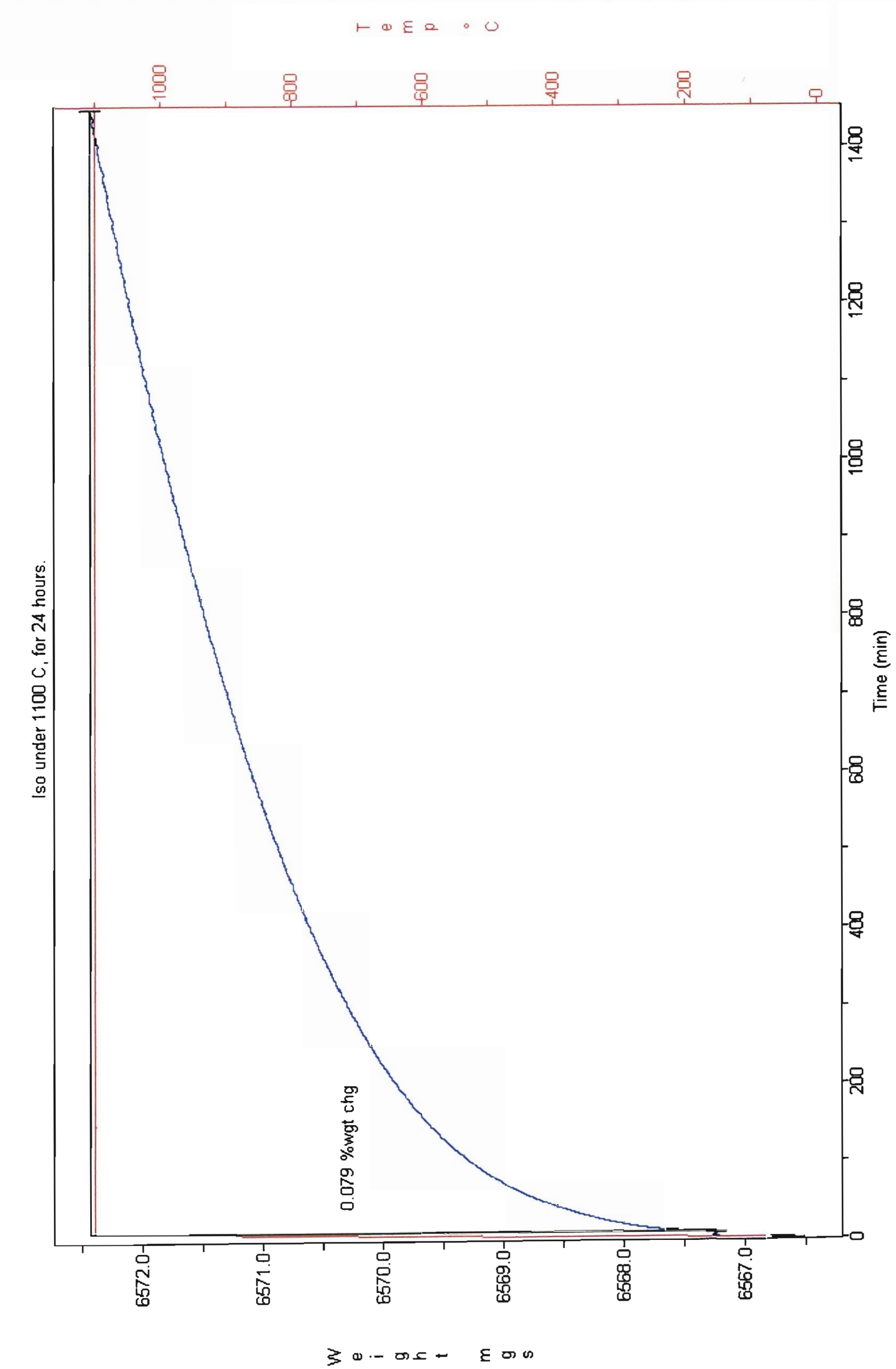
Figure 1. Sample piece run under 1100 °C for 24 hours.




Total
wt gain
5.2mg
Area≈6.7cm²
ΔW/A
≈0.78 mg/cm²

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Project No. 06002.01.081
Book No. 1
TITLE 850°C - Air - 24hr

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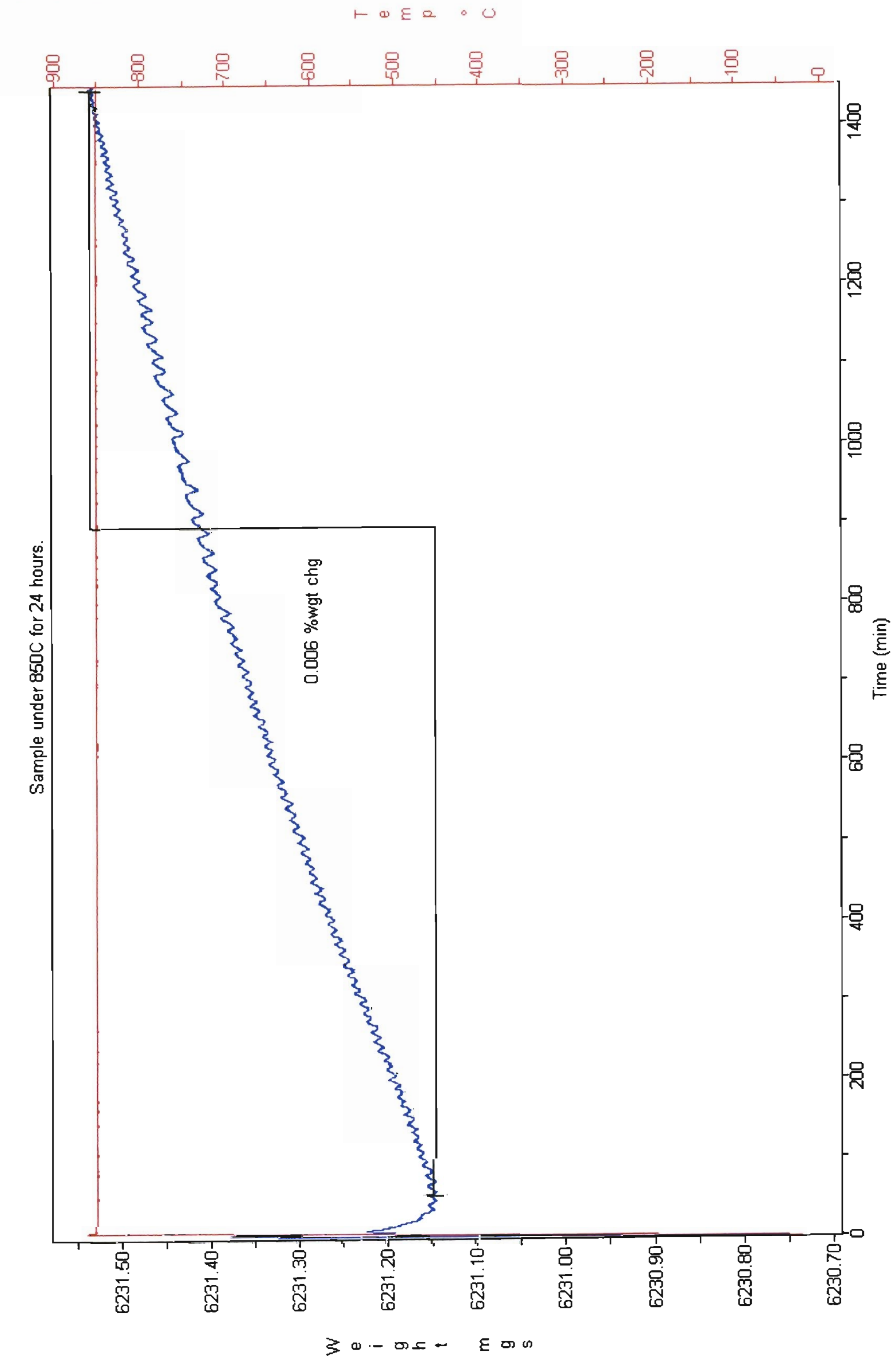
Figure 2. Sample piece run under 850 °C for 24 hours.




wt change
= 6231.53
− 6231.15
0.38 mg

0.38/6.7 cm²

0.06 mg/cm²

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Project No. 06002.01.081
Book No. 1
TITLE 600°C - Air - 48 h

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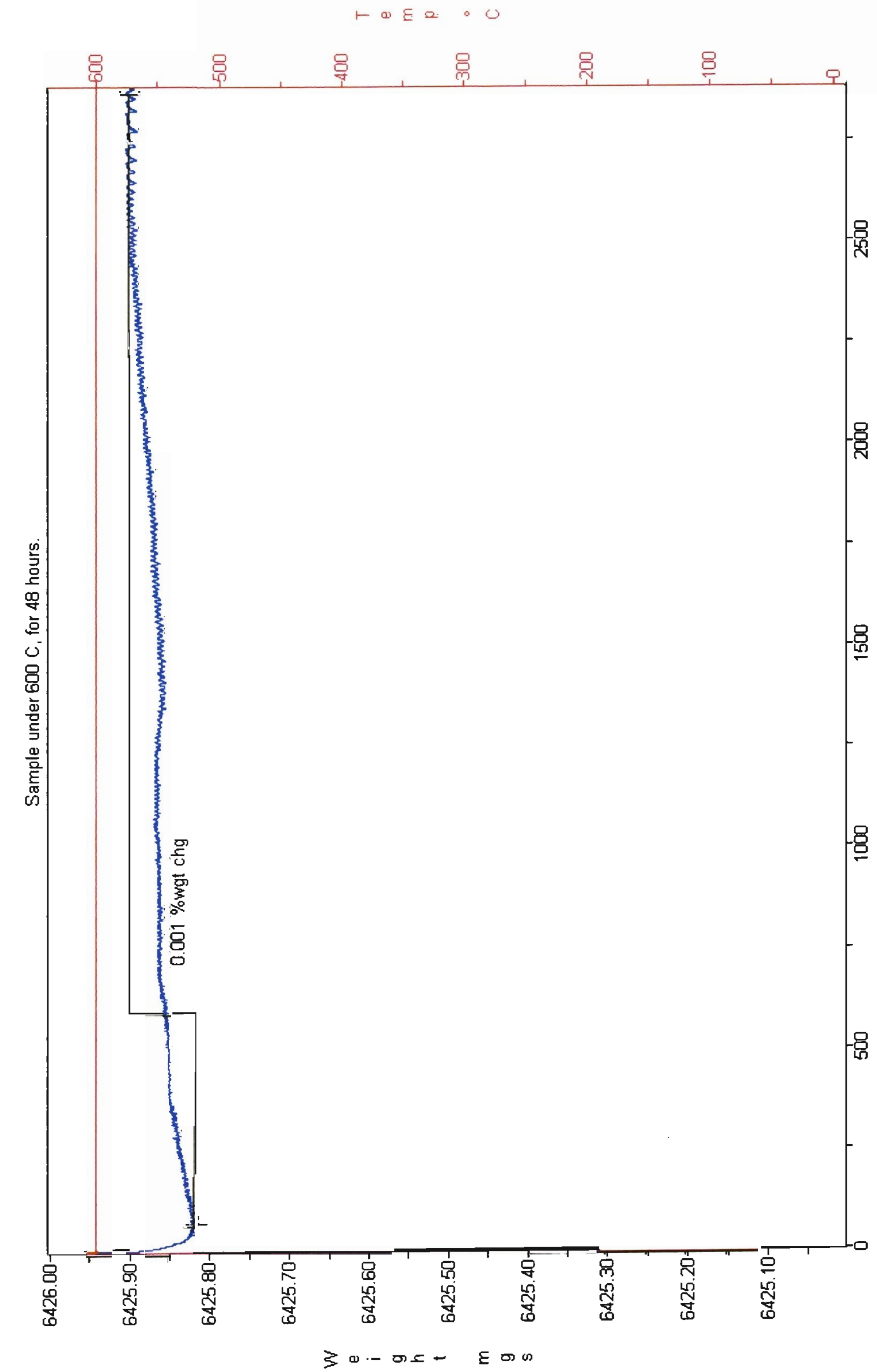
Figure 3a. Sample piece run under 600 °C for 48 hours.

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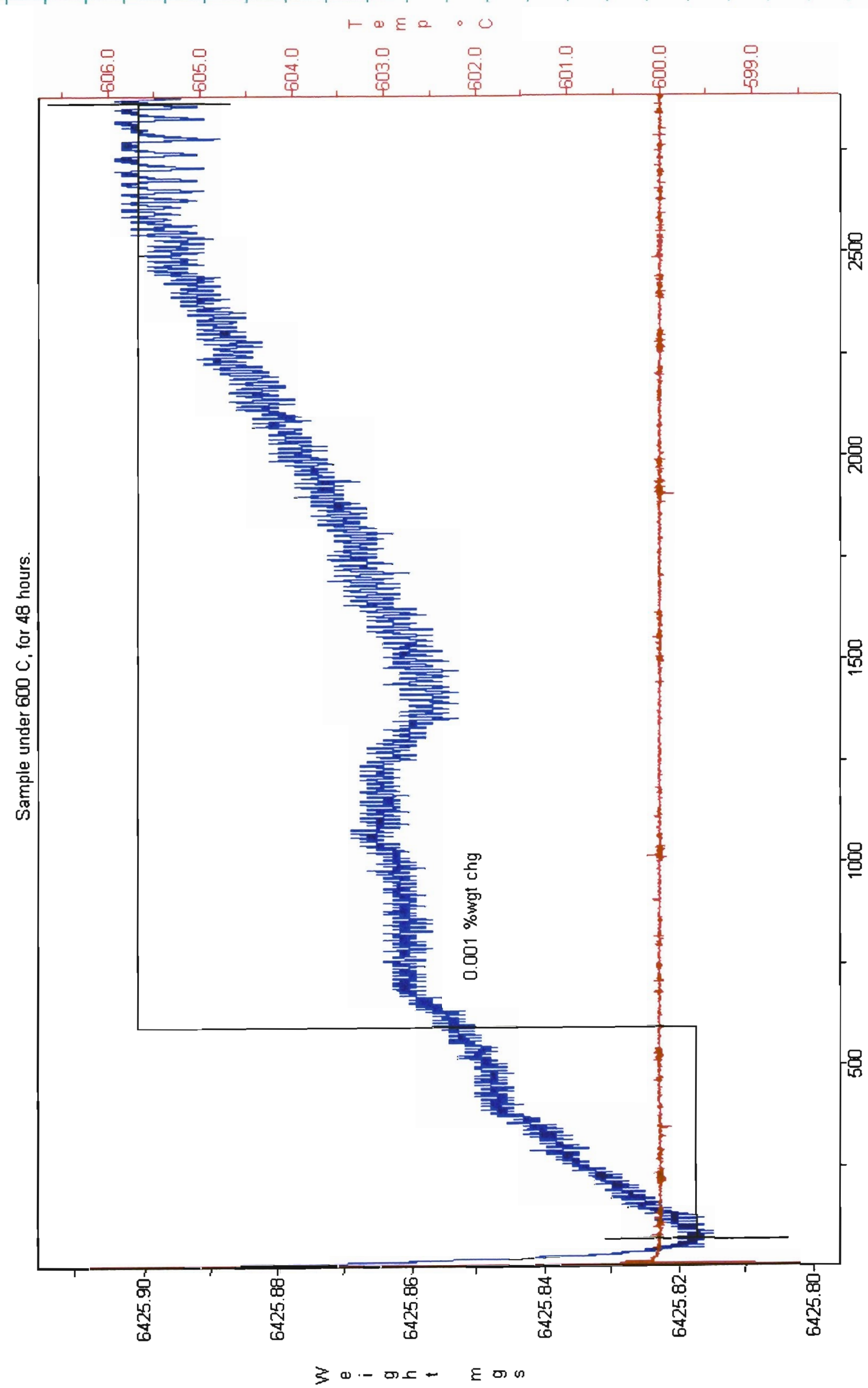
|---|---|---|---|
| | | Recorded by K. J. Chiang | 12/10/03 |

Project No. 06002.01.081
Book No. 1
TITLE 600°C - Air - 48h

From Page No.

Figure 3b. Sample piece run under 600 °C for 48 hours, zoomed in plot.

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| | | Recorded by K.T. Ching | 12/10/03 |

TITLE 350°C - Air - 48h
Project No. 06002.01.081
Book No. 1



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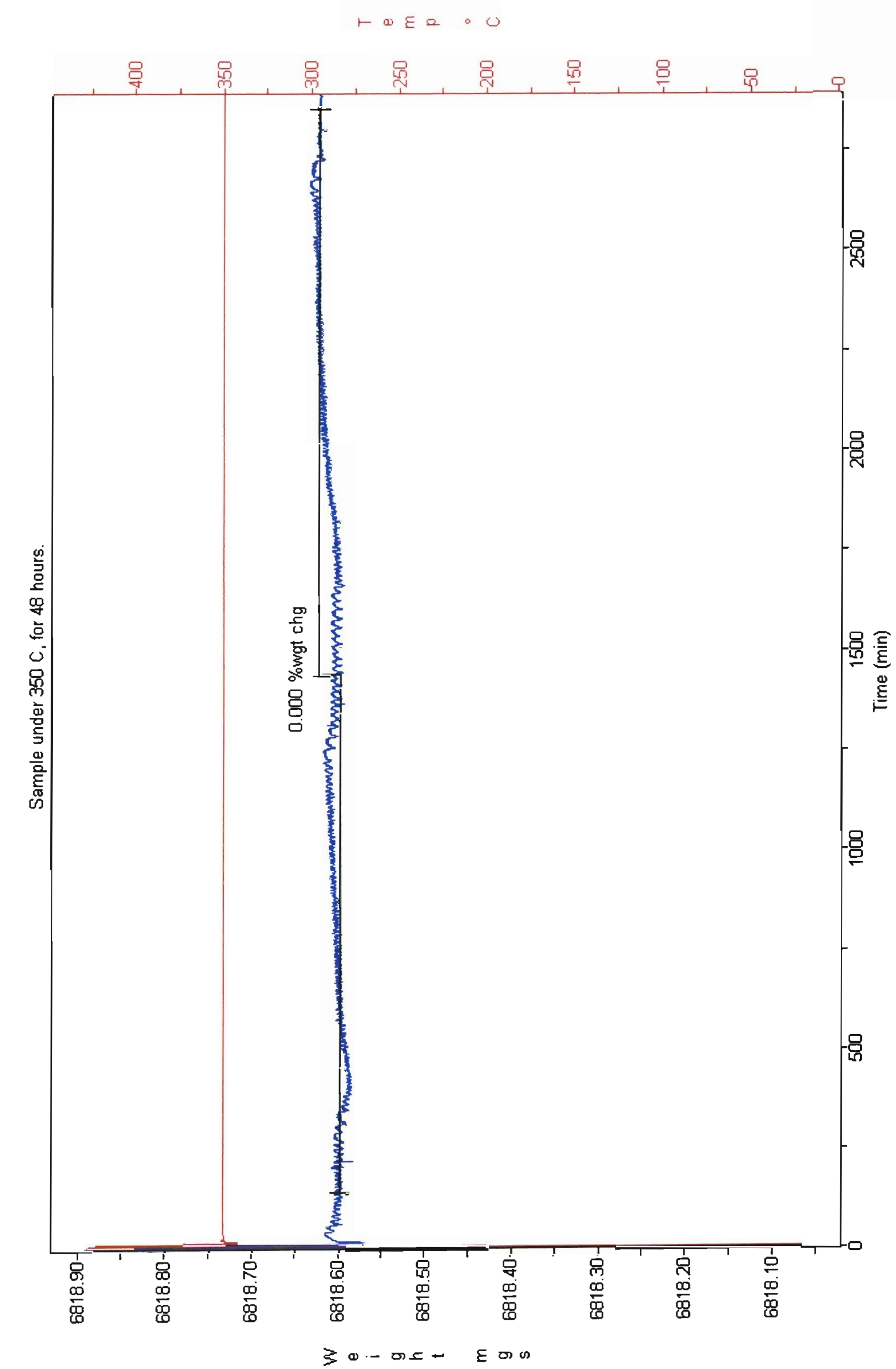
Figure 4a. Sample piece run under 350 °C for 48 hours.

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Book No. 1
TITLE 350°C - Air - 48h

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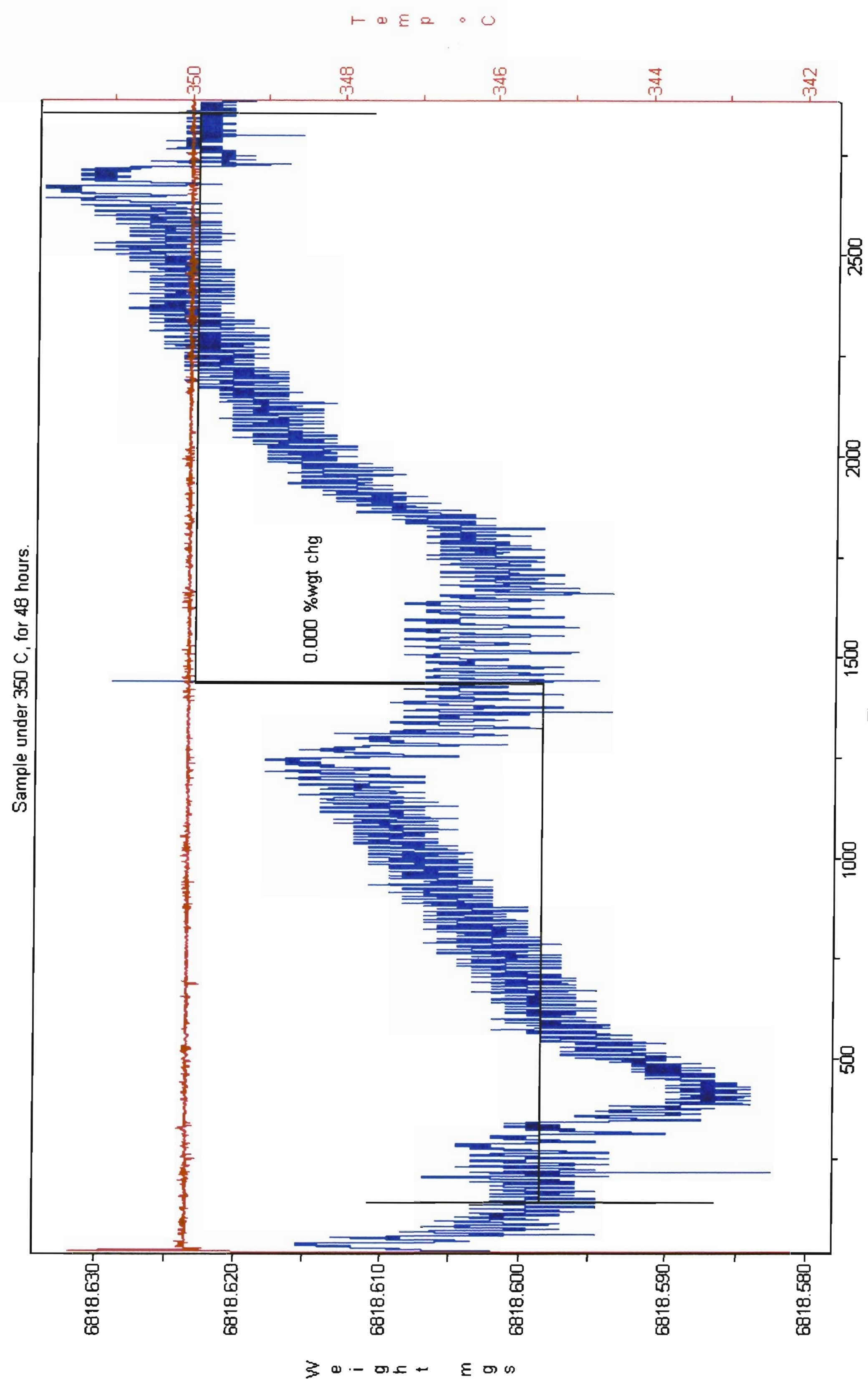


Figure 4b. Sample piece run under 350 °C for 48 hours, zoomed in plot.

To Page No. ___

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TITLE Thermogravimetric Analysis (TGA) unit
Project No. 06002.01.081
Book No. 1



From Page No. ___

Versatherm 100 g, 1 µg. 1100°C

Thermax 1500°C .1 µg 1.5 g

1700°C max 1 µg 100 gm

Brochure

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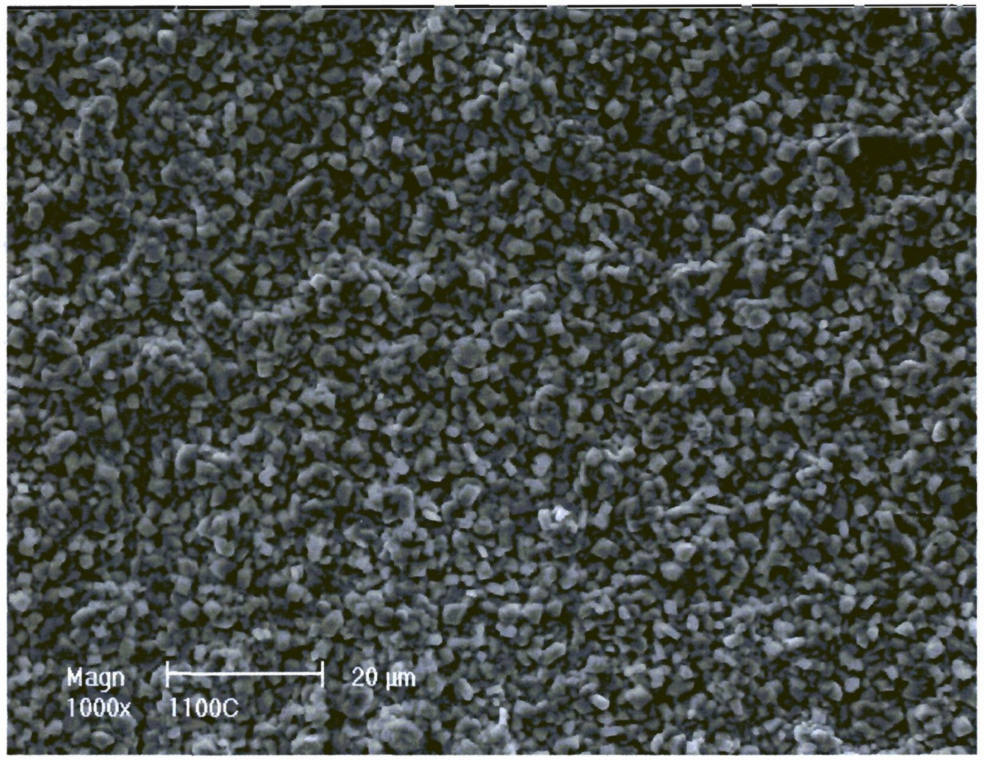
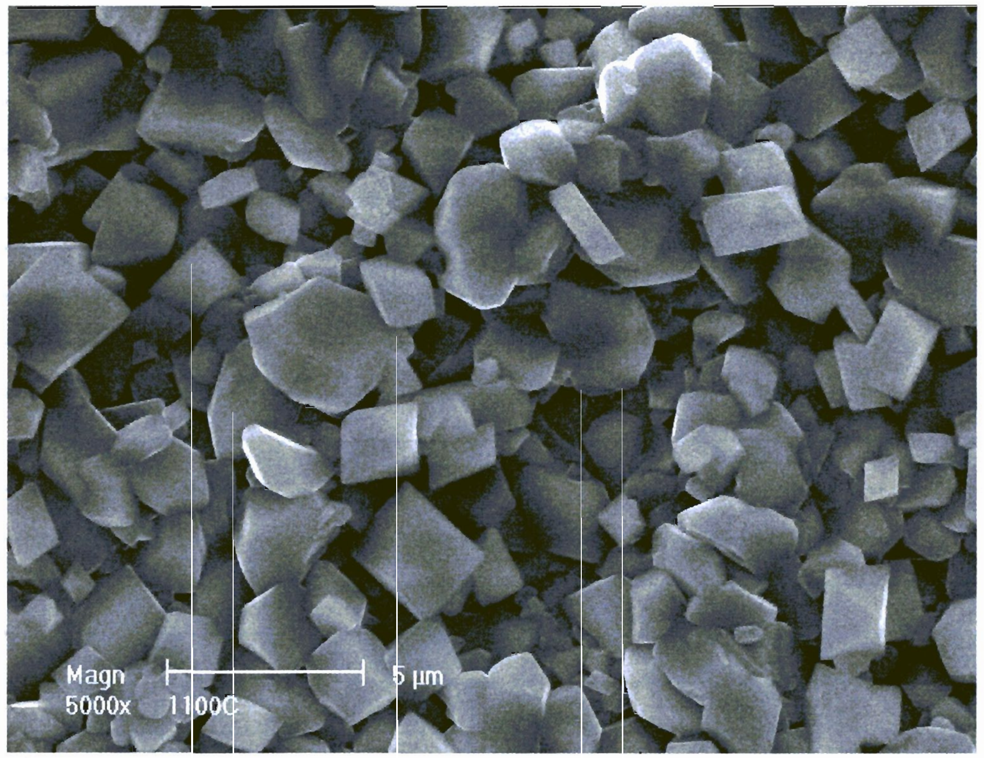
Project No. 06002.01.081
Book No. 1 TITLE SEM 1100°C - Air - 24h Sample

From Page No.

1100°C - 24 hrs

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TITLE EDX, XRD 1100°C - Air - 24h
Project No. 06002.01.081
Book No.

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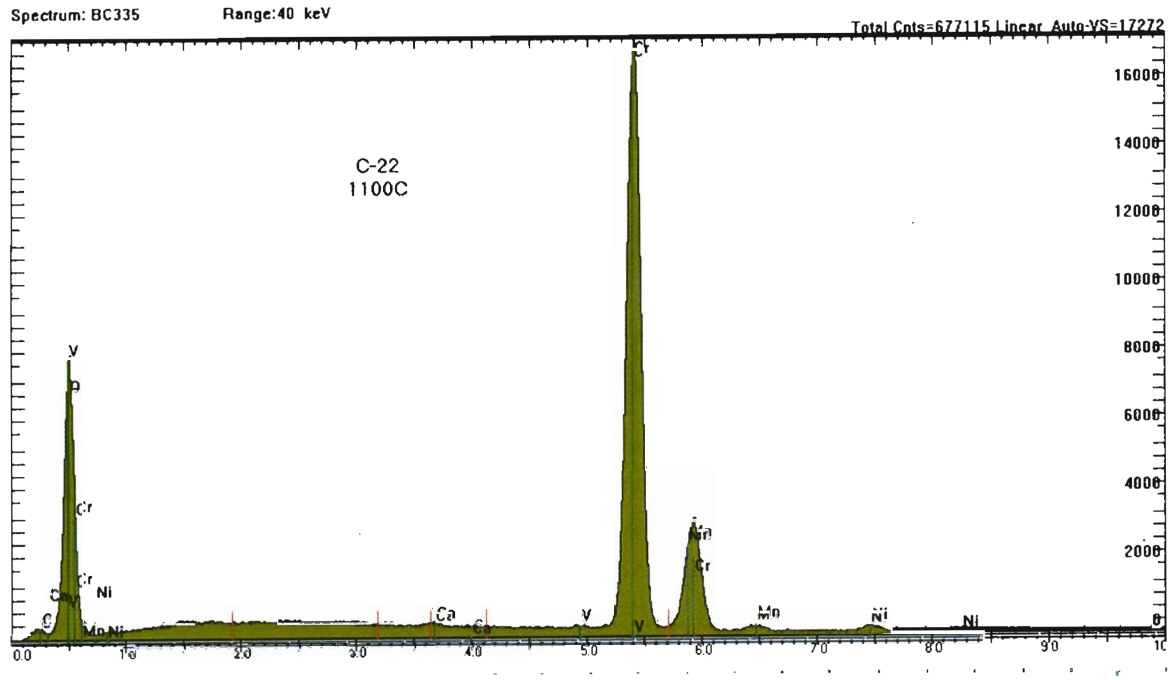


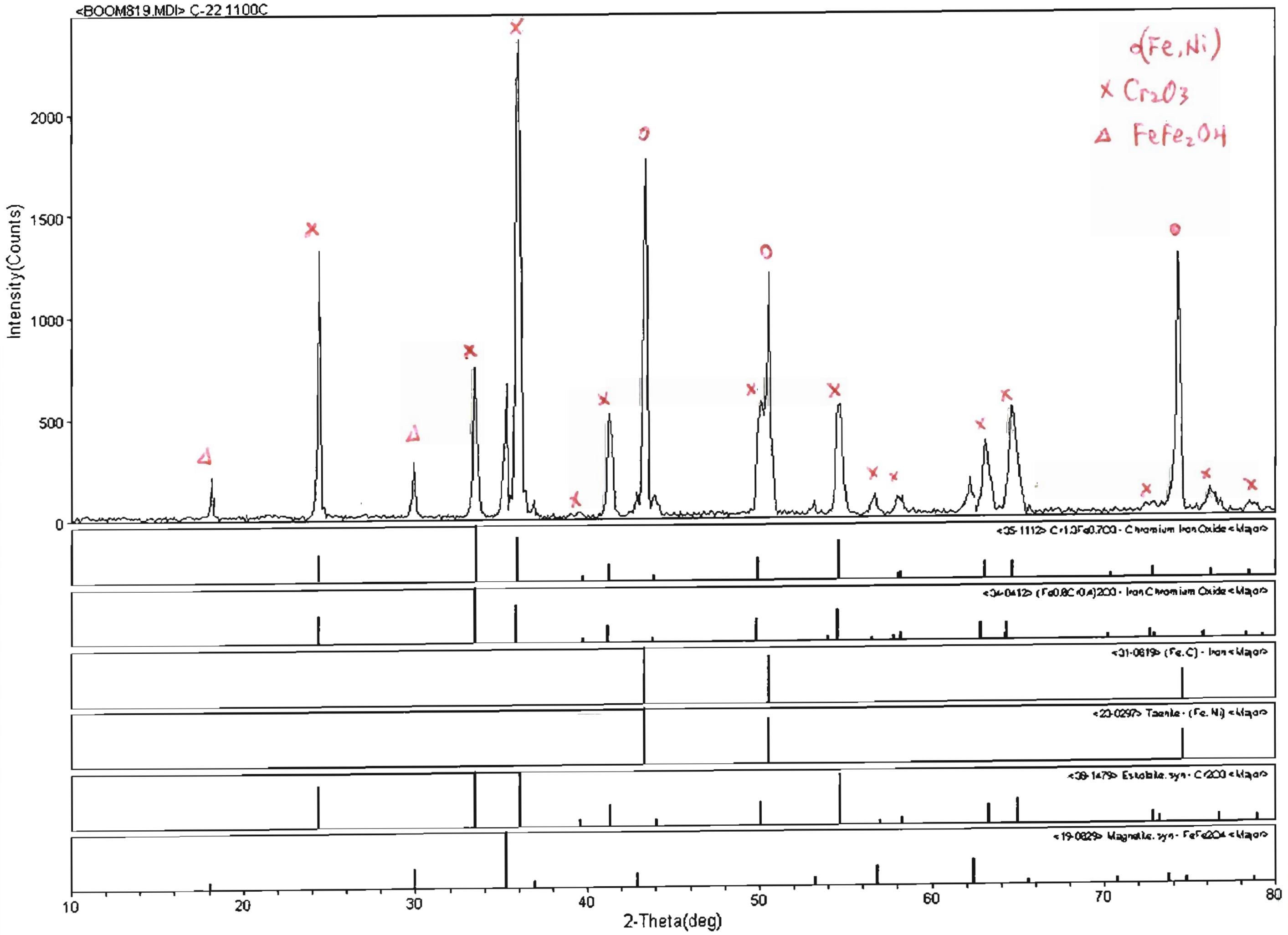


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Project No. 06002.01.081
Book No. 1
TITLE SEM 850°C - Air - 24hr

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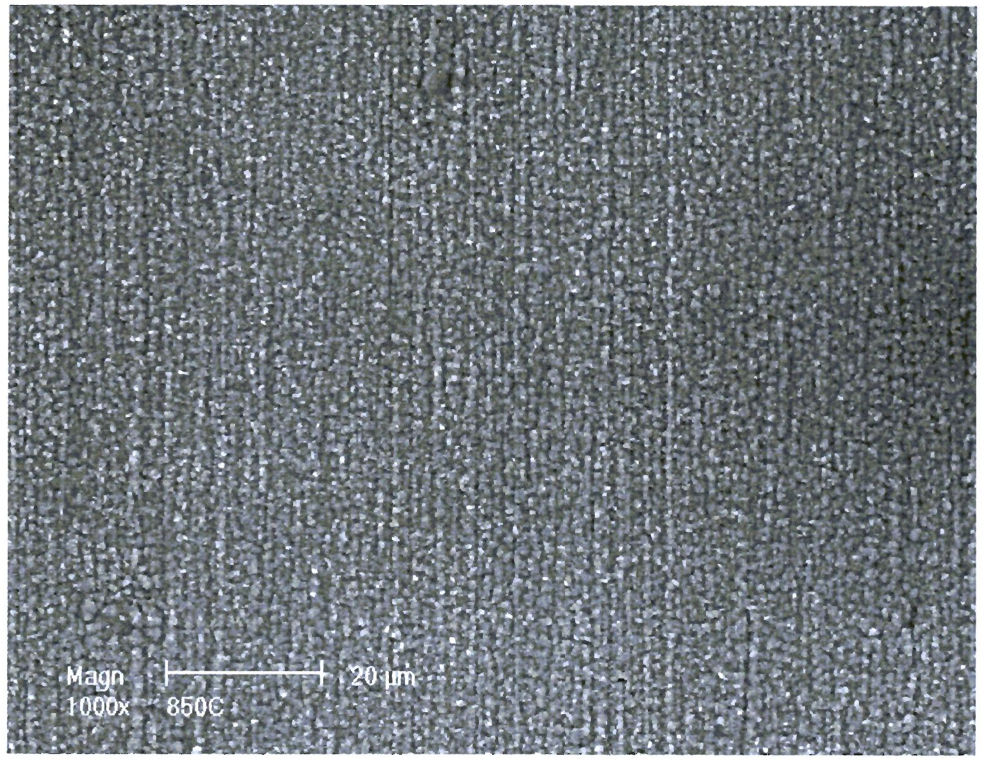
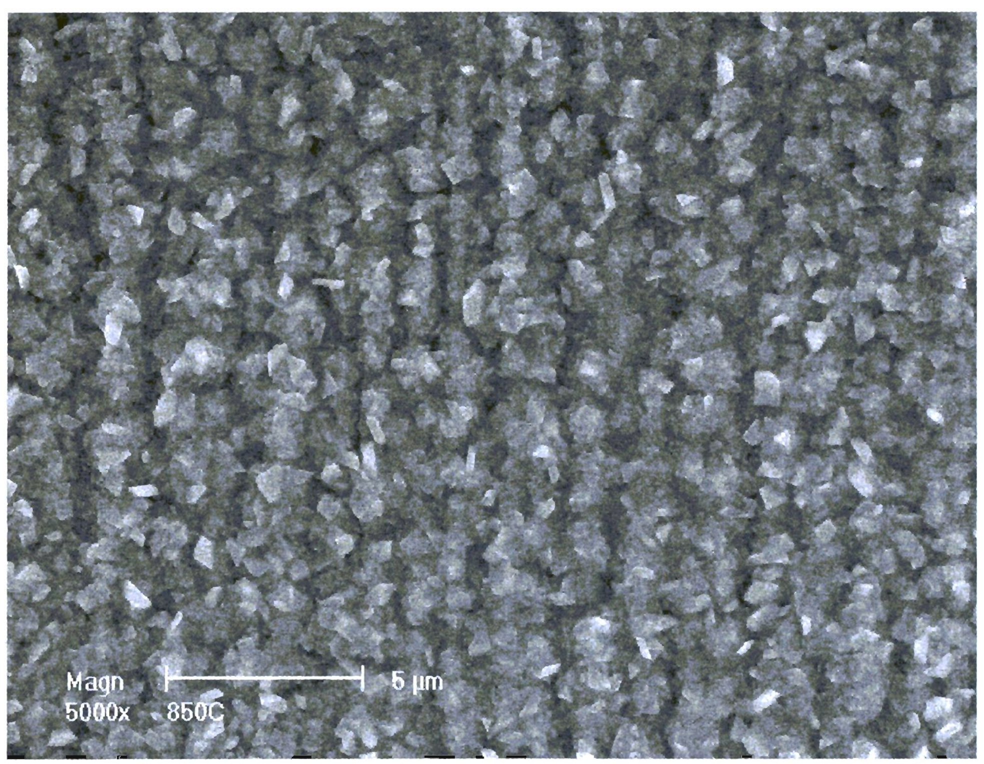
850°C - 24hrs

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Book No. 1
TITLE EDX, XRD 850°C - Air - 24h

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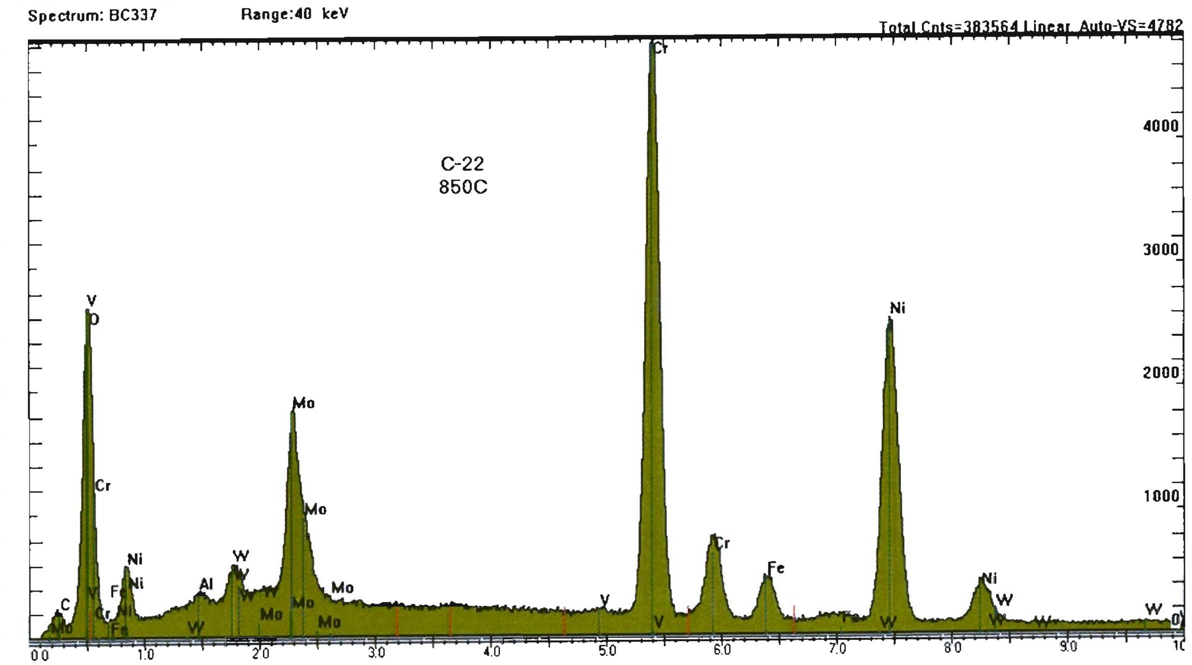


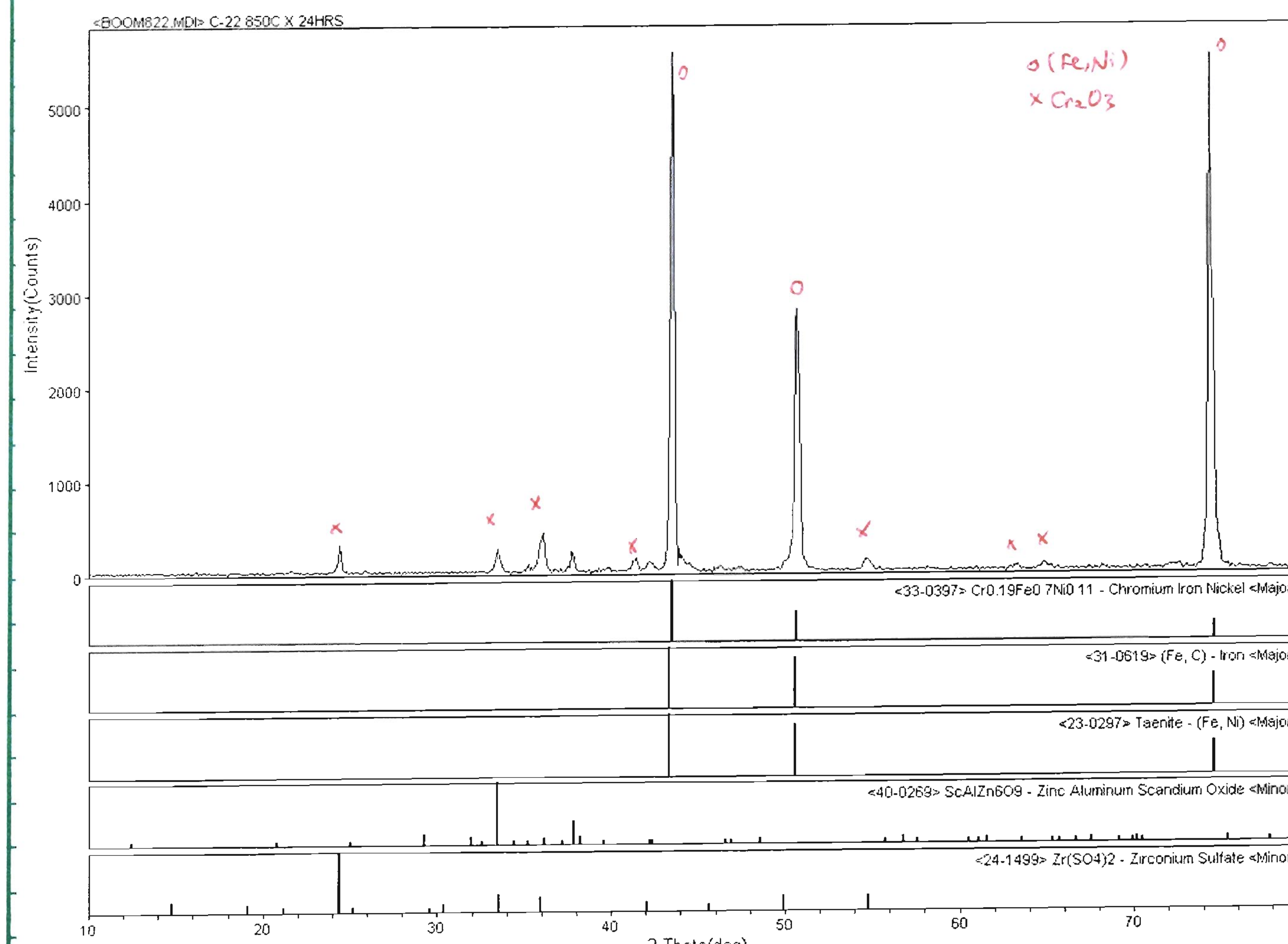


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Project No. 06002.01.081
Book No. 1 TITLE SEM 600°C - Air - 48h

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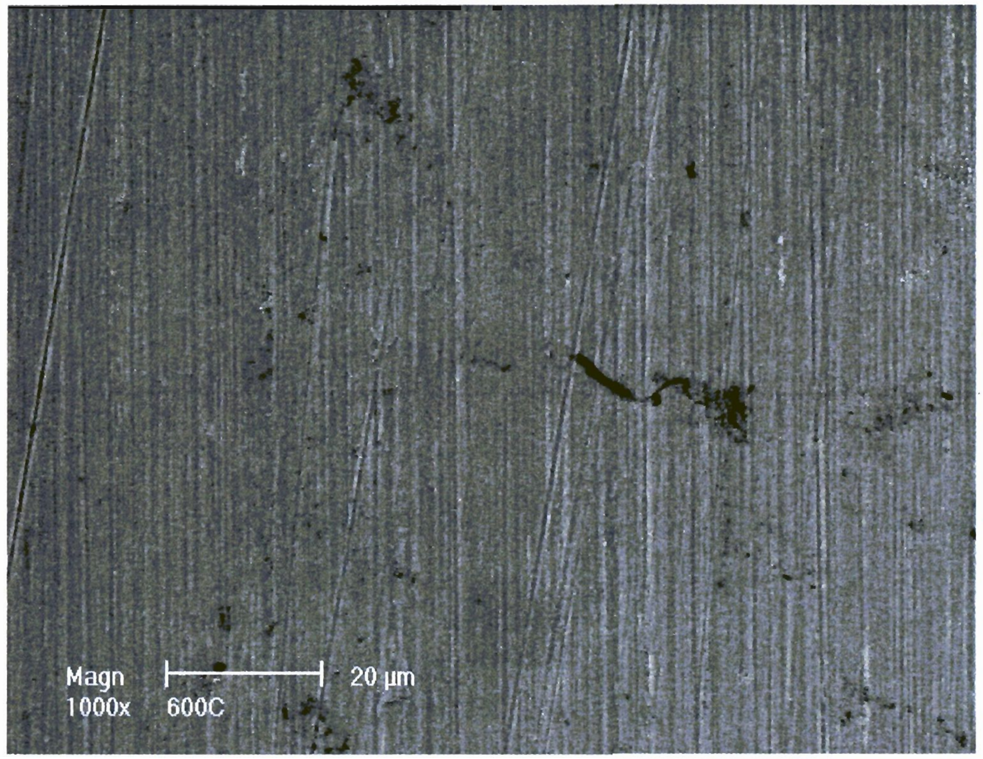
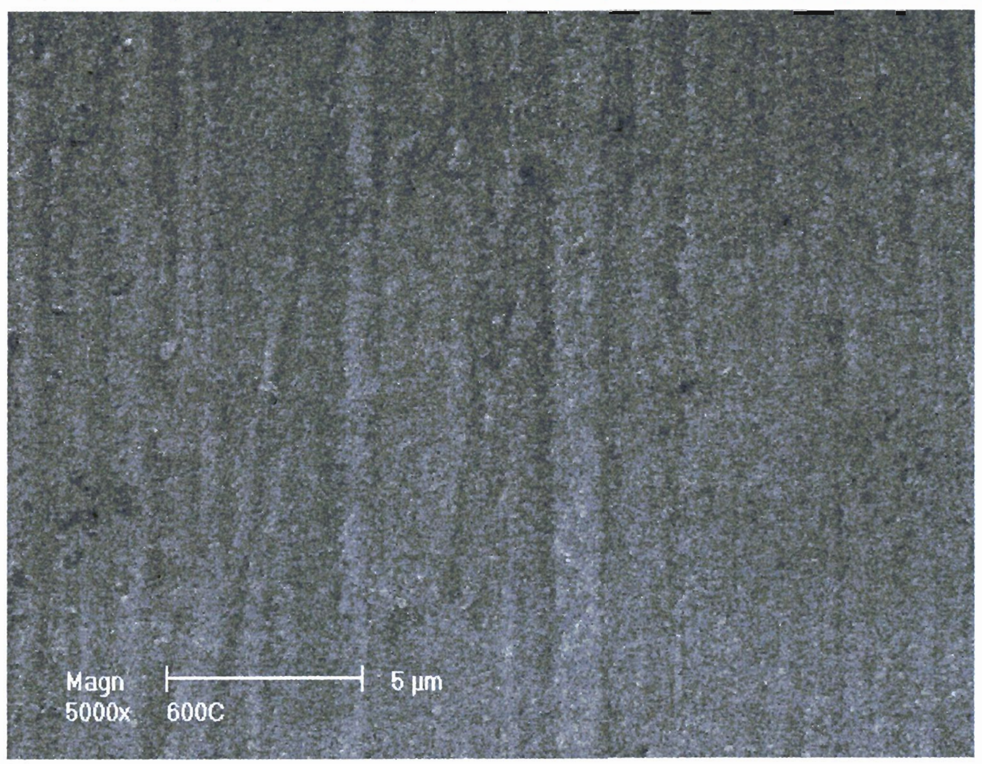
600°C-48h

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TITLE EDX, XRD 600°C - Air - 48h
Project No. 06002.01.081
Book No. 1

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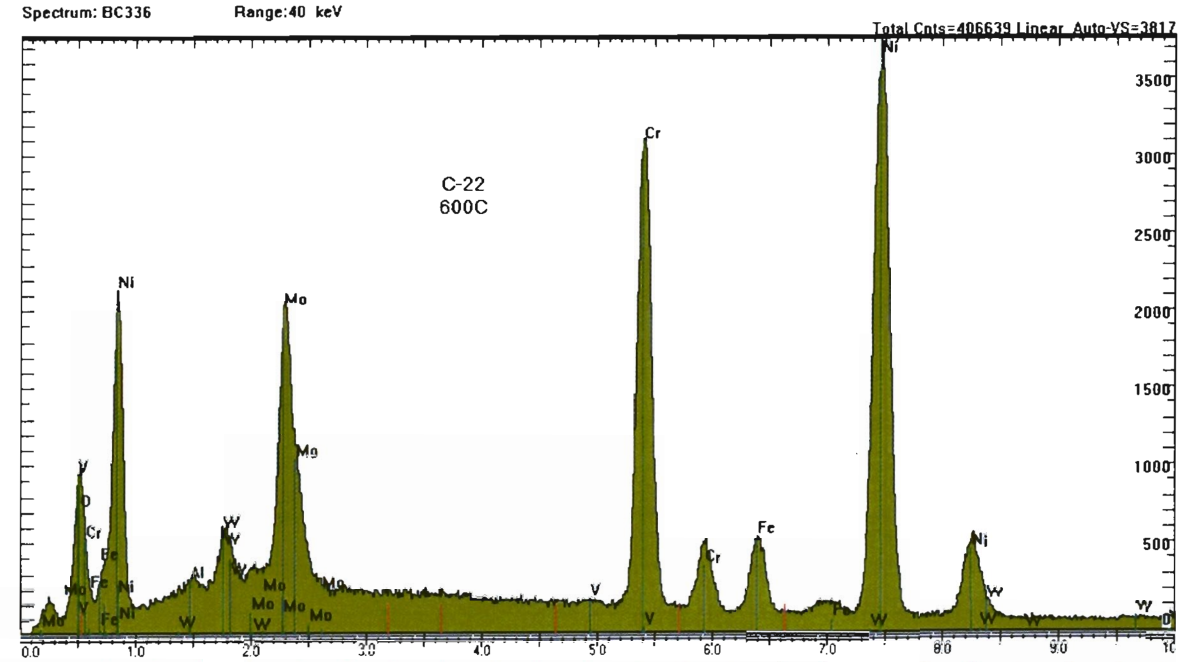


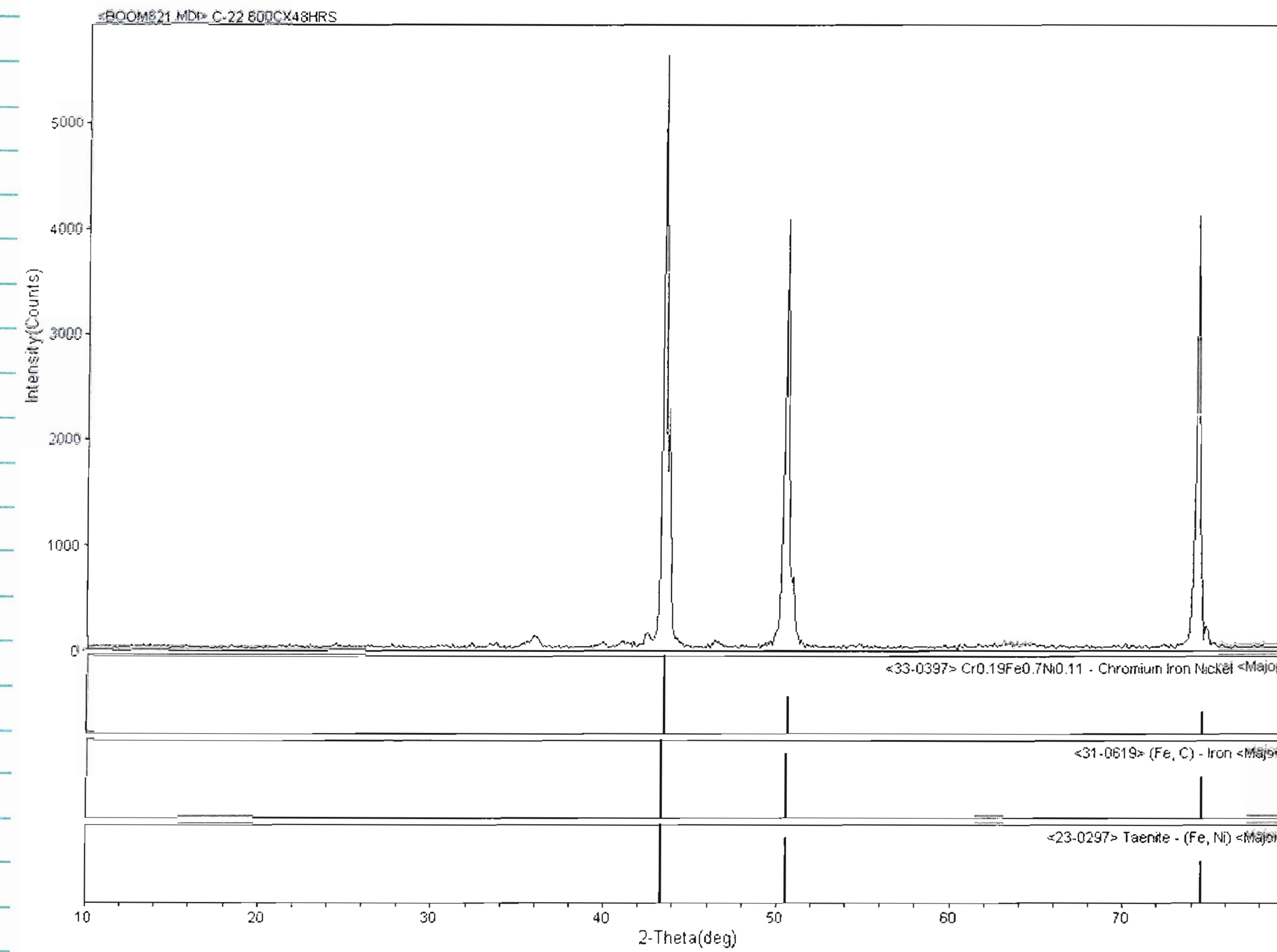


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TITLE SEM 350°C-48h-Air

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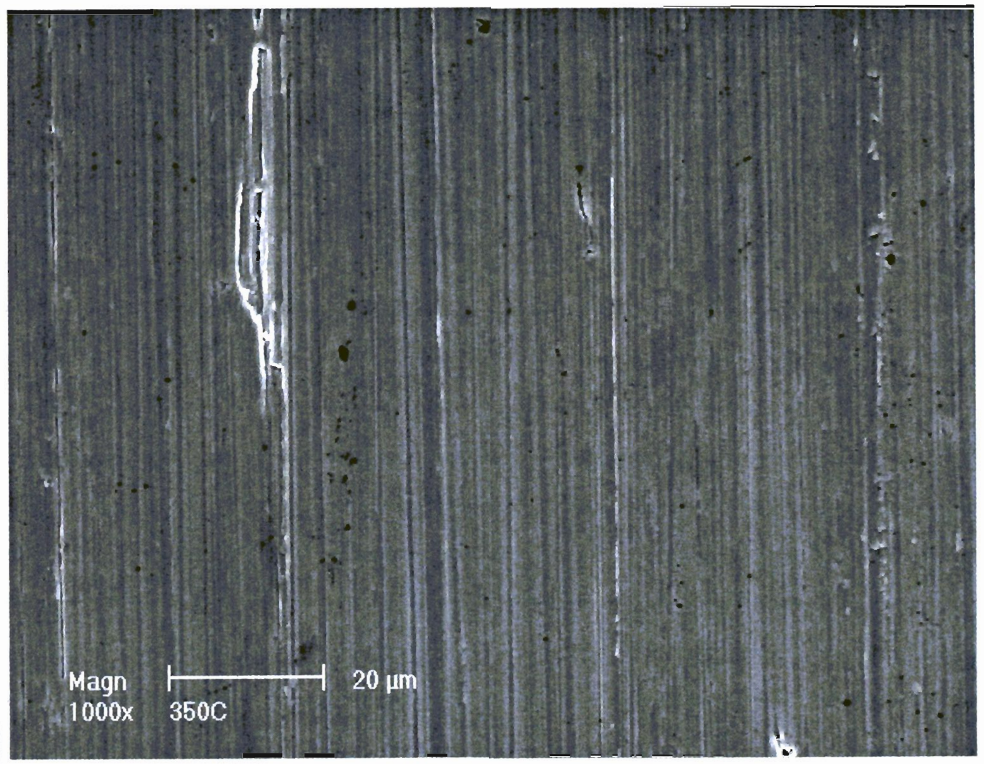
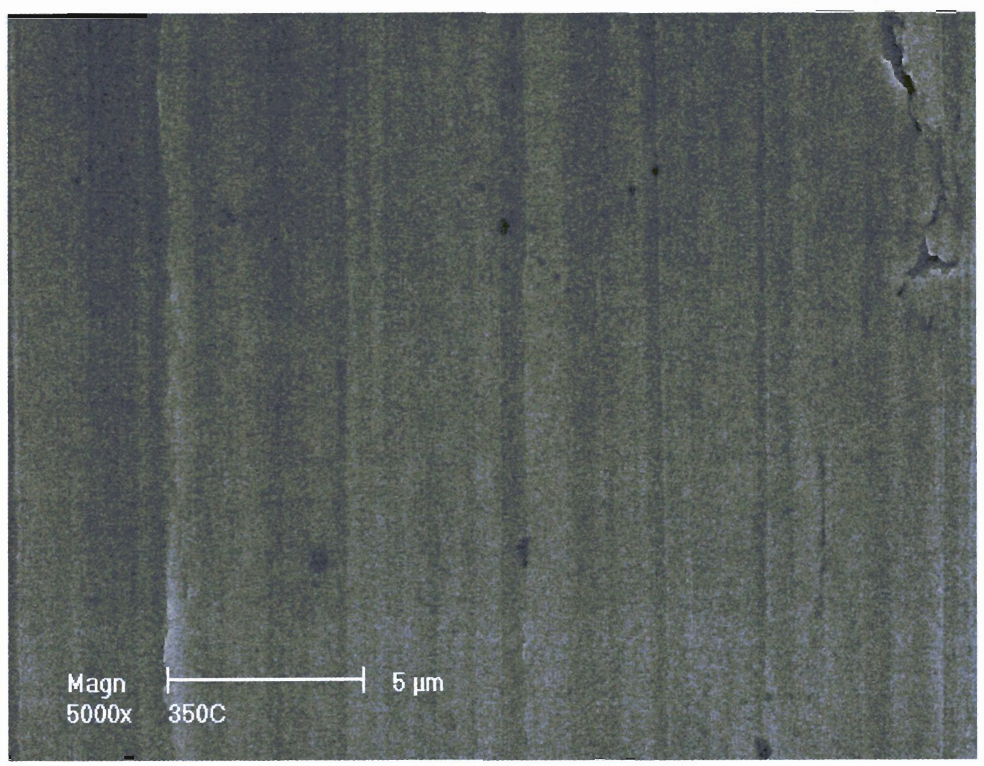
350°C
-48hrs

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TITLE EDX, XRD 350°C-48h-Air
Project No. 06002.01.081
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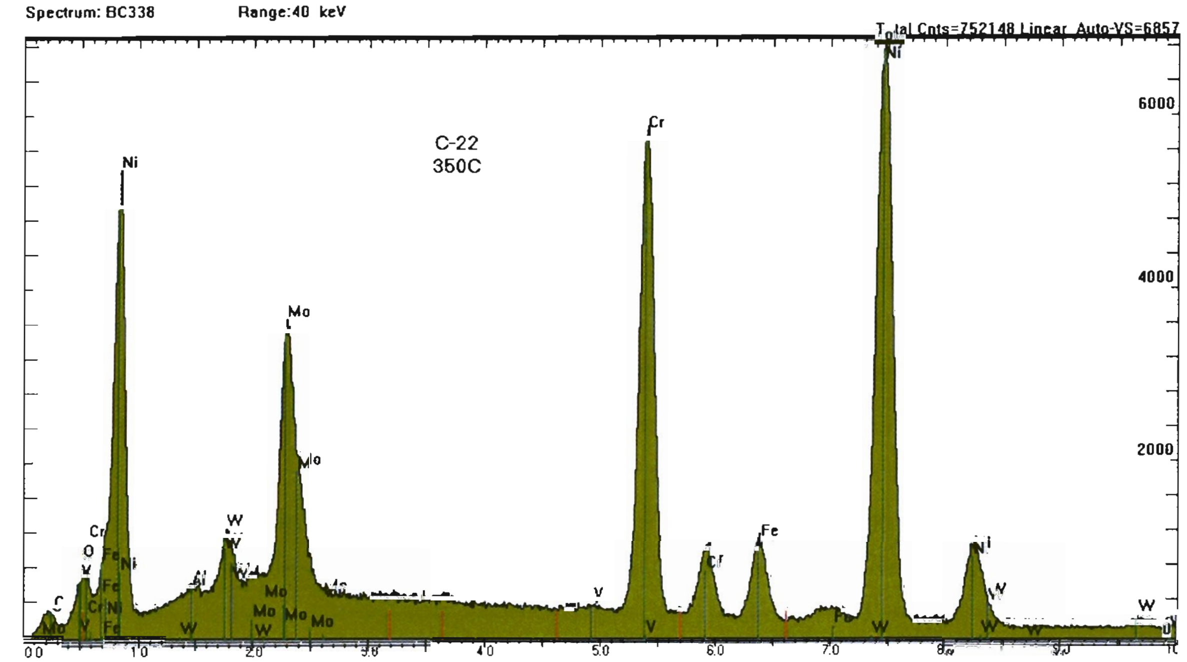


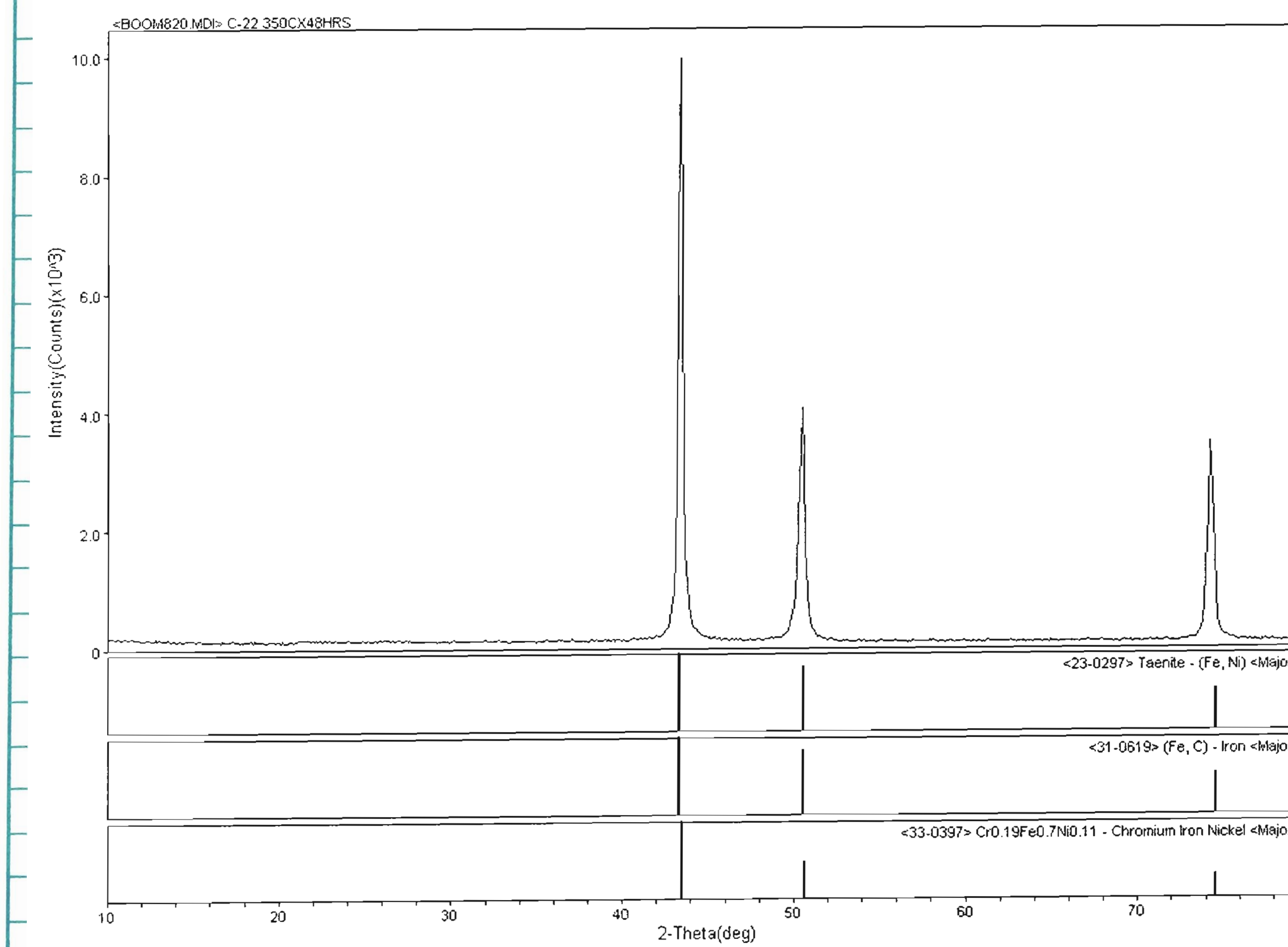


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From Page

ICON

Icon Services Inc., Laboratory, PO BOX 220, Mt. Marion, NY 12456

Fax / Phone: (845)246-1802

## *Certificate of Analysis*

Technical Data Information

**Product Name: Oxygen Gas-$^{18}O_2$** **Date: February 17, 2004**

**Lot No: CH-021604-1** **Cat No: IO 6393**

**Isotopic Purity**: Based on Mass. Spect.:

$^{18}O = 99.9$atom%; $^{16}O < 0.1$atom%; $^{17}O = 0.1$atom%

**Chemical Purity**: 99.9%, Based on GC:

$N_2 < 15$ppm; $CO_2 = 86$ppm;

$(CH_4 + C_2H_6) < 30$ppm

Cheng-hua Lee, Chemist I

*ICON SERVICES INC.*
*STABLE ISOTOPES AND LABELED COMPOUNDS FOR RESEARCH*

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|---|---|---|---|
| | | Recorded by K. J. Chiu | 2/19/04 |



From Page

ICON SERVICES, INC.
OLD KINGS HWY.
MT. MARION., NY 12456
TEL. 1-845-246-1802

MATERIAL SAFETY DATA SHEET

SECTION 1. ---------- CHEMICAL IDENTIFICATION----------
CATALOG #: IO6393
NAME: OXYGEN-18, 99 ATOM% O18
SECTION 2. ----- COMPOSITION/INFORMATION ON INGREDIENTS ------
CAS #: 7782-44-7
MF: O2
EC NO: 231-956-9
SECTION 3. ---------- HAZARDS IDENTIFICATION ---------
LABEL PRECAUTIONARY STATEMENTS
OXIDIZING
CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE.
DANGER: HIGH-PRESSURE OXIDIZING GAS.
KEEP AWAY FROM COMBUSTIBLE MATERIAL.
WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.
SECTION 4. ---------- FIRST-AID MEASURES----------
IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS. CALL A PHYSICIAN.
IF INHALED, REMOVE TO FRESH AIR. IF BREATHING BECOMES DIFFICULT, CALL A PHYSICIAN.
IN CASE OF SKIN CONTACT, FLUSH WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES. REMOVE CONTAMINATED CLOTHING AND SHOES. CALL A PHYSICIAN.
IN CASE OF CONTACT WITH EYES, FLUSH WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES. ASSURE ADEQUATE FLUSHING BY SEPARATING THE EYELIDS WITH FINGERS. CALL A PHYSICIAN.
SECTION 5. --------- FIRE FIGHTING MEASURES ----------
EXTINGUISHING MEDIA
CARBON DIOXIDE, DRY CHEMICAL POWDER OR APPROPRIATE FOAM.
SPECIAL FIREFIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS
VIGOROUSLY SUPPORTS COMBUSTION.
EMITS TOXIC FUMES UNDER FIRE CONDITIONS.
CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE.
MAY ACCELERATE COMBUSTION.
CONTAINER EXPLOSION MAY OCCUR UNDER FIRE CONDITIONS.
SECTION 6. -------- ACCIDENTAL RELEASE MEASURES--------
ABSORB ON SAND OR VERMICULITE AND PLACE IN CLOSED CONTAINERS FOR DISPOSAL.
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

To Page No.

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| | | Recorded by K. J. Chiu | 2/19/04 |

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SECTION 12. - - - - - - - - - - ECOLOGICAL INFORMATION - - - - - - - - - -
DATA NOT YET AVAILABLE.
SECTION 13. - - - - - - - - - - DISPOSAL CONSIDERATIONS - - - - - - - - -
CAUTION: NO-RETURN CYLINDER. DO NOT REUSE. EMPTY CYLINDER WILL CONTAIN HAZARDOUS RESIDUE. FOLLOW PROPER DISPOSAL TECHNIQUES.
CONTACT A LICENSED PROFESSIONAL WASTE DISPOSAL SERVICE TO DISPOSE OF THIS MATERIAL.
OBSERVE ALL FEDERAL, STATE AND LOCAL ENVIRONMENTAL REGULATIONS.
SECTION 14. - - - - - - - - - - TRANSPORT INFORMATION - - - - - - - - -
CONTACT ICON SERVICES, INC FOR TRANSPORTATION INFORMATION.
SECTION 15. - - - - - - - - - - REGULATORY INFORMATION - - - - - - - - - -
EUROPEAN INFORMATION
EC INDEX NO: 008-001-00-8
OXIDIZING
R 8
CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE.
S 17
KEEP AWAY FROM COMBUSTIBLE MATERIAL.
REVIEWS, STANDARDS, AND REGULATIONS
OEL=MAK
NOHS 1974: HZD M0006; NIS 23; TNF 917; NOS 33; TNE 9677
NOES 1983: HZD M0006; NIS 81; TNF 7113; NOS 90; TNE 189129; TFE 87473
EPA GENETOX PROGRAM 1988, POSITIVE: V79 CELL CULTURE-GENE MUTATION
EPA TSCA SECTION 8(B) CHEMICAL INVENTORY
EPA TSCA SECTION 8(D) UNPUBLISHED HEALTH/SAFETY STUDIES
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, OCTOBER 2000
NIOSH ANALYTICAL METHOD, 1994: OXYGEN, 6601
SECTION 16. - - - - - - - - - - OTHER INFORMATION- - - - - - - - - - - -
THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE, ICON SERVICES, INC SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITIONS OF SALE.


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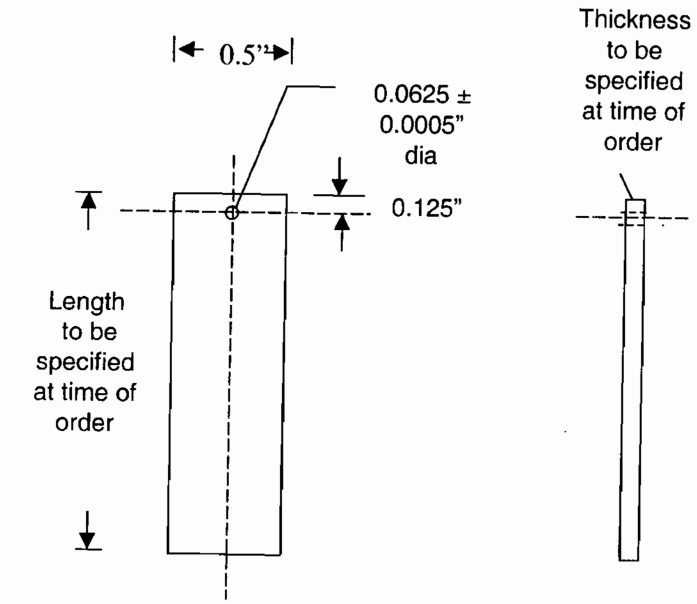
From Page No.

Specimen Design for Dry oxidation Test of C22 alloy

| Ken Chiang<br>SwRI-CNWRA<br>Phone: (210) 522-2308<br>Fax: (210) 522-5184<br>e-mail: kchiang@swri.org | Deliquescence Specimen<br>CNWRA 20.06002.01.081.010<br>All Dimension ± 0.005"<br>unless otherwise specified<br>Revision 0 | To be completed at time of order:<br>Material: ____<br>Heat: ____<br>Specimen Orientation: ____<br>Other: ____ |
| --- | --- | --- |




K. T. Chiang 2/6/04
Initiated by K. Chiang Date

2/6/04
Reviewed by V. Jain Date

2/6/04
QA Approval R. Brient Date

0.6" x 0.50" sample machined from C22 Heat 2277-3-3266.

To Page No.

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| --- | --- | --- | --- |
| | | Recorded by K. T. Chiang | 3/22/04 |

From Page No.

## Kuang-Tsan Ken Chiang

**From:** Forrest Campbell [fcampbell@swri.edu]
**Sent:** Monday, March 22, 2004 10:29 AM
**To:** Kuang-Tsan K Chiang
**Cc:** Sastry Cheruvu
**Subject:** Last Specimen Finished at 1100C

div-20 cyclic.xls

See attached excel file. The Last Specimen #6, 1100C 100 hours has a loose powdery oxide.
What do I do now? Furnace is still at 1100C, do you want me to shut it down until we have the other
large specimen group? If so then I have to make another specimen holder at the machine shop to hold the specimens.

Forrest

Forrest S. Campbell Sr. Eng. Technologist
Southwest Research Inst
6220 Culebra Rd
San Antonio, Texas 78238
Phone : (210)522-5647 or (210)522-2322
Fax : (210)522-6220
---

Div 20 20.06002.01.081

| Specimen # | Length(in.) | Width(in) | Thick(in) | urface area(in^ | tial weight | hal Weight | weight change % | Cycles | Temp C |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 1.997 | 0.5000 | 0.06260 | 2.30962 | 8.62138 | 8.62315 | 0.02053 | 10 | 870 |
| 4 | 2.000 | 0.4985 | 0.06060 | 2.29682 | 8.45353 | 8.45675 | 0.03809 | 100 | 870 |
| 5 | 1.997 | 0.4970 | 0.06230 | 2.29577 | 8.61926 | 8.63220 | 0.15013 | 10 | 1100 |
| 6 | 1.992 | 0.4985 | 0.06170 | 2.29279 | 8.46370 | 8.27519 | -2.22728 | 100 | 1100 |

To Page No.

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|---|---|---|---|
| | | K. T. Chiang | 3/22/04 |



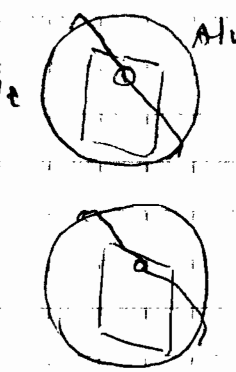
From Page No. C22 Sample machined from 2277-3-3266 Heat

Place samples in furnace at 650°C in air
For 2 weeks test

#1 Dimension 14.40mm × 12.69 mm × 1.56 mm
Initial wt 2.43026 g
Final wt 2.43031 g (2wk)
Visual light grey, shiny

#2 Dimension 14.71 × 12.56 × 1.47 mm
Initial WT 2.28552
Final wt 2.28560 (2wks)

#2 Inside



Alumina crucible
Sample holds by Ni-Cr wire

Sample in 4/29/04 12:56pm

#1

2wks out 5/13/04 12:56pm

Used Lindberg oven Model # 51333 SN# 909172
oven set point: 650°C

oven Temperature - 657.4°C
Measurement Taken with Omega Microprocessor Thermometer model # HH22
SN# T-94140 cal 4/27/04 due 10/27/04
Thermocouple # 330 cal 1/22/04 due 7/22/04

weight measurements Taken with Sartorius Genius Scale
SN# 12809099 cal 11/14/03 due 5/14/04

Cycle 2 Sample back in 5/13/04 13:30 pm

To Page No.

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|---|---|---|---|
| | | [signature] | 5/13/04 |

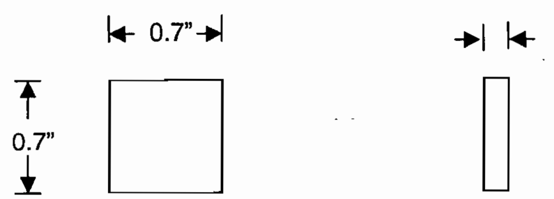
From Page No.

Drawing for passive film characterization

Ken Chiang
SwRI-CNWRA
Phone: (210) 522-2308
Fax: (210) 522-5184
e-mail: kchiang@swri.org

Passivity SIMS Specimen
CNWRA 20.06002.01.081.011
All Dimension ± 0.01"
unless otherwise specified

To be completed at time of order:
Material:
Heat:
Specimen Orientation:
Other:




K. J. Chiang 5/14/04
Initiated by K. Chiang Date

[signature] 5/14/04
Reviewed by V. Jain Date

[signature] 5/14/04
QA Approval R. Brient Date

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| | | Recorded by K. J. Chiang | 5/14/04 |



From Page No.

Cycle 2 Sample out 650°C
5/17/04 15:00 pm 2wks 12hr

Sample 2 2.28553 g

Sample 1 2.43026 g

Cycle 3 Sample in
5/17/04 15:40 pm
Sample out
6/16/04 9:40 am 29 days 18 hrs 650°C

Sample 1 2.43024 g 2.43025 g

Sample 2 2.28554 g , 2.28580 g

Cycle 4 6/23/04 3:40 pm in
7/1/04 1:40 pm out 7 days 22 hrs 650°C
Sample 1 2.43020 2.43019

Sample 2 2.28550

Cycle 5 7/1/04 2:20 pm in
7/8/04 2:20 pm out
Sample 1 2.43019
Sample 2 2.28550

Cycle 6 7/11/04 5:20pm in 2.28548 g
7/18/04 5:20pm out 2.43016 g

Cycle 7 7/18/04 5:30 pm in 2.28548 g
7/25/04 5:30 pm out 2.430[illegible] g

Cycle 8 7/27/04 8:30 Am in 2.28545 g
8/18/04 7:30 Am out 2.43014 g

To Page No.

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|---|---|---|---|
| | | Recorded by K. J. Chiang | 8/18/04 |

From Page No.

Message Page 1 of 1

## Kuang-Tsan Ken Chiang

**From:** Kuang-Tsan Ken Chiang [kchiang@cnwra.swri.edu]
**Sent:** Tuesday, June 29, 2004 8:49 AM
**To:** 'CHEN, DUN'
**Cc:** 'Eubanks, Jeff'
**Subject:** TGA Specimens

Dun,

Thank you for the information on delivery. Congratulation for your promotion.

Under a separate cover, I am sending you (3) C22 alloy samples for TGA testing.

Sample Description- C22 alloy specimens, approximate dimension 15 mmx 13mm x1.4 mm, weight 2.3 gram.
Please run TGA in slow flowing air
1) 1100C-120 hrs, 2) 850C -120 hrs, 3) spare, or 950C-120 hrs

Expect Results- parabolic oxidation kinetics. Please provide plot and numerical data -First hour every 10 min, subsequent data point every hour.
Heating profile- Maximum heting rate to temperature, keep constant temperature during exposure.

Please return all specimens.

Thank you and best regards,

Ken

Ken Chiang, Ph. D.
Senior Research Scientist
Corrosion Science & Process Engineering
Southwest Research Institute
6220 Culebra Road
San Antonio, Texas 78238-5166
(210) 522-2308
Fax: (210) 522-5184
kchiang@swri.org

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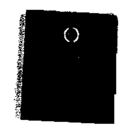
| | | Recorded by K.T. Chiang | 9/10/04 |



From Page No.

① 1100°C - AIR - 5 days (120 hours)
14.61mm × 12.62mm × 1.45 mm
2.26776 g
Fisherbrand®

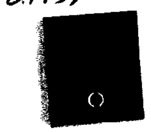
② 850°C - AIR - 5 days (120 hours)
14.68 mm × 12.67 mm × 1.43 mm
2.25469 g
Fisherbrand®



Area = 2(1.844 + 0.212 + 0.183) − 2(π(1/32 × 2.54)²) + 2πr(0.145)
= 4.478 − 0.040 + 0.072
= 4.51

Area = a − b + c

Area = 2(1.468 × 1.267 + 1.468 × 0.143 + 1.267 × 0.143)
= 4.502
b = −0.040
c = 2π 1/16 1/2 × 2.54 × l
= 0.5 × 0.143 = 0.072
a + b + c = 4.534



a = 2(1.452 × 1.264 + 1.452 × 0.152 + 1.264 × 0.152)
= 4.496
b = −0.040
c = 0.5 × 0.152 = 0.076
Area = 4.532

③ Spare 950°C - 5 days
14.52mm × 12.64mm × 1.52mm
0.572 × 0.4975 × 0.060
2.38492 g
Fisherbrand®

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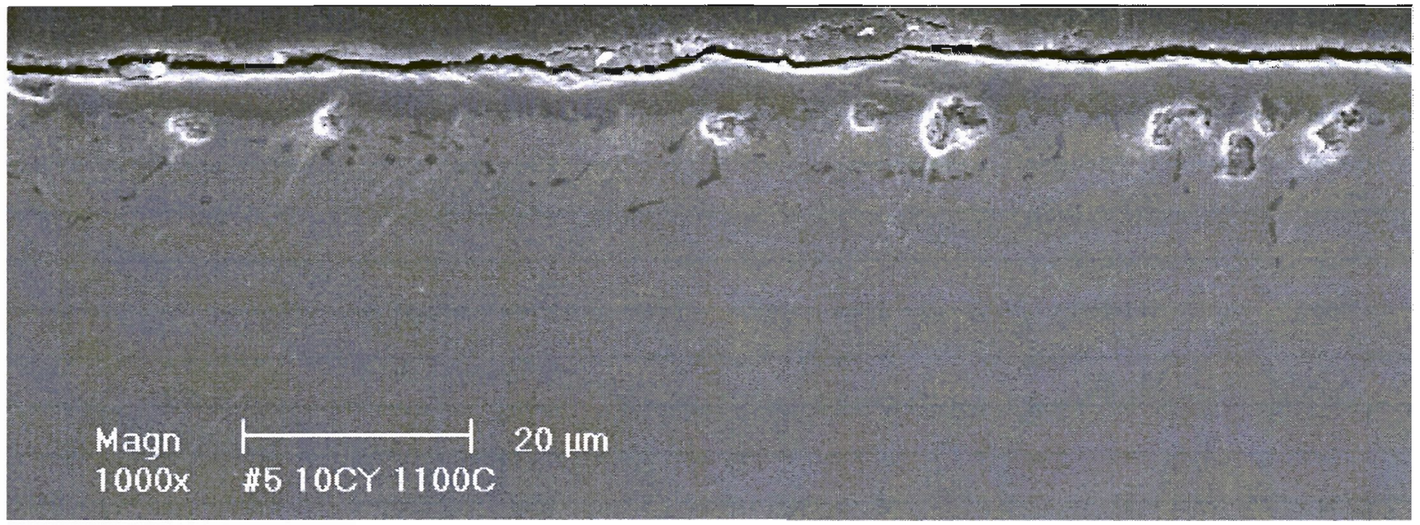
Project No. 06002.01.081
Book No. 1
TITLE Cyclic Oxidation

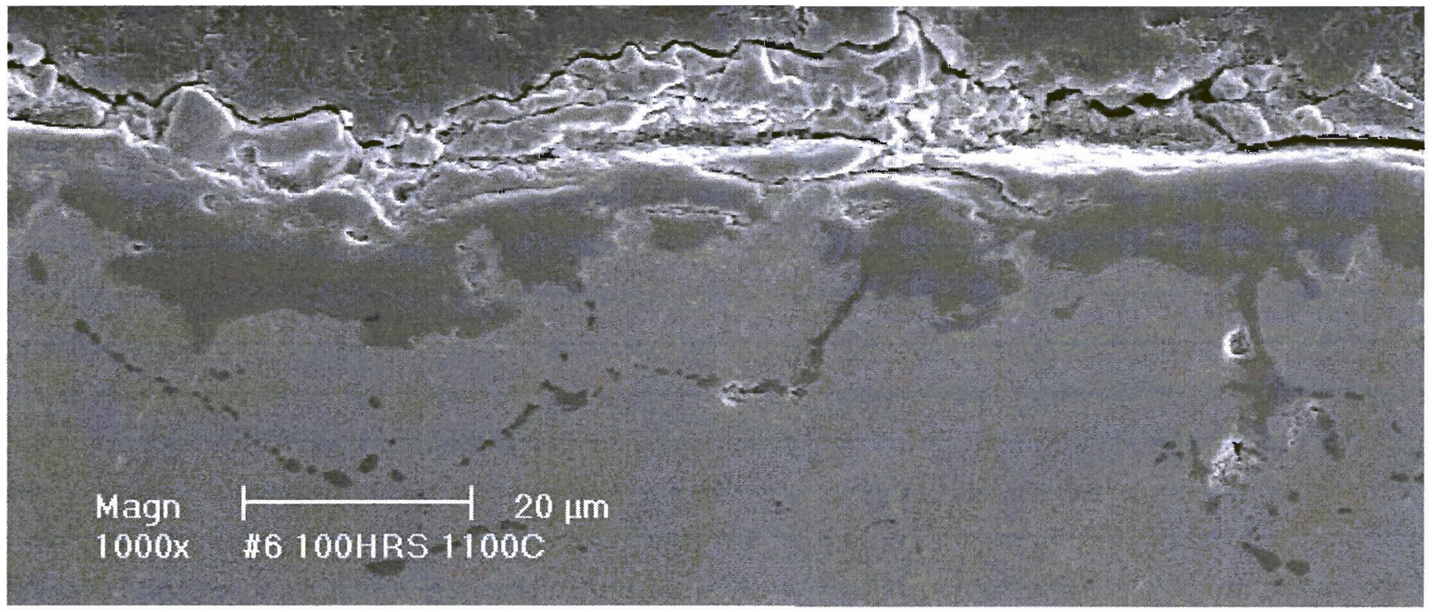
From Page No.

Alloy 22

1100°c 10 cycles




1100°c 100 cycles

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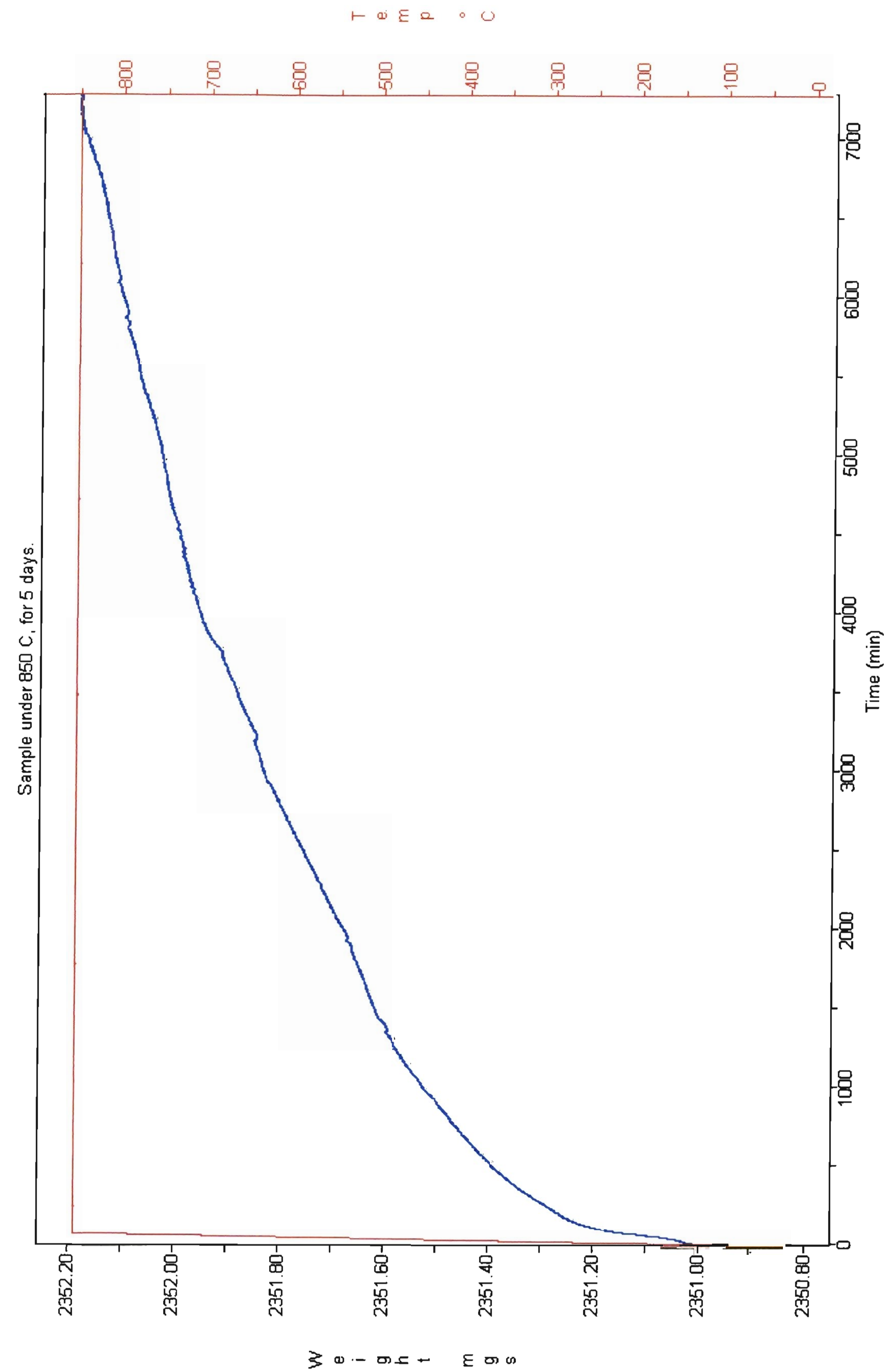
| | | K.T. Cheng | 9/10/04 |

Project No. 06002.01.321
TITLE TGA
Book No. 1


From

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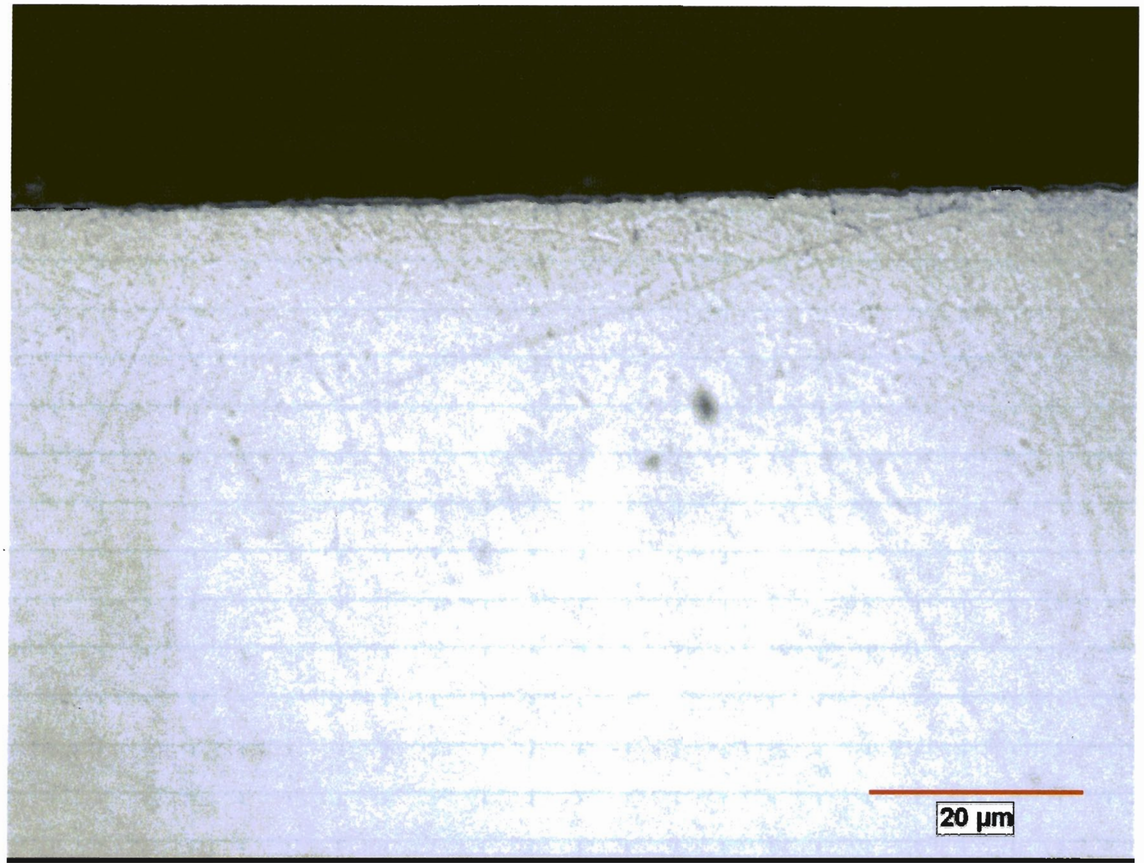
| --- | --- | --- | --- |
| | | K.T. Cheng | 10/14/2004 |

Project No. 06002.01.321

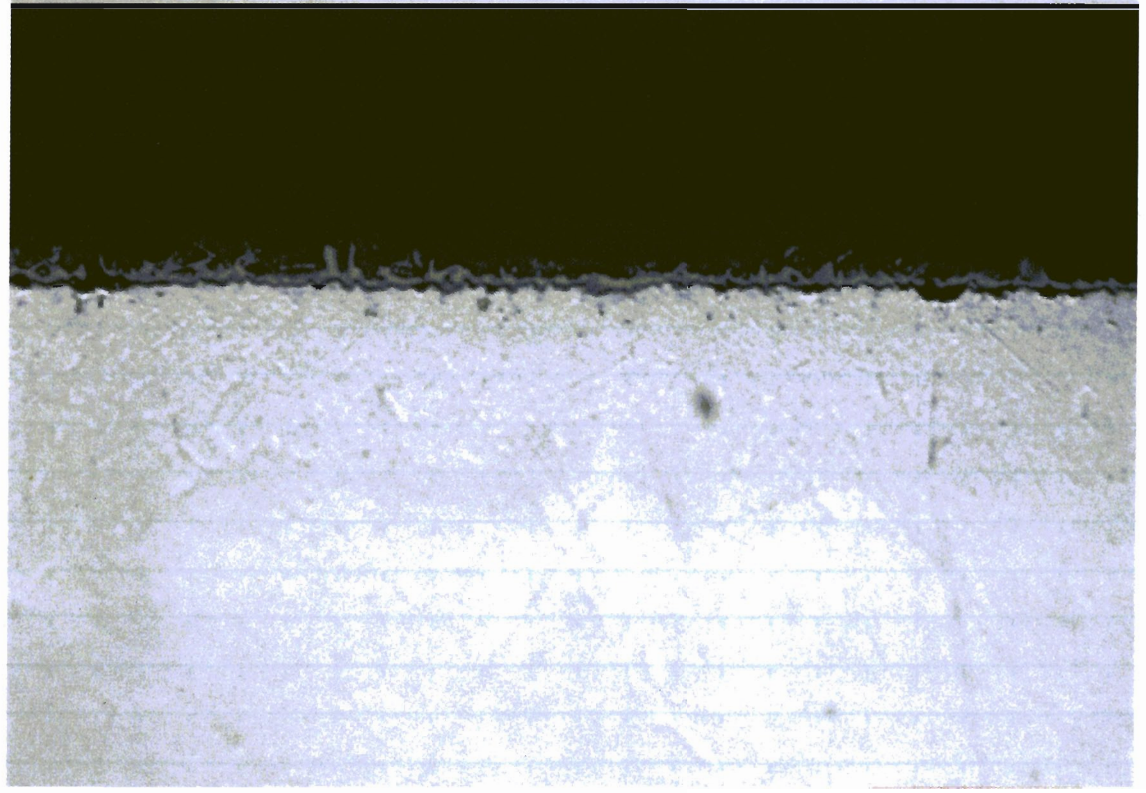
Book No. 1 TITLE Optical Metallography

From Page No.

850°C - AiR - 24 h




850°C
Air
120h



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TITLE Surface SEM

Project No. 06002.01.321

Book No. 1



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Project No. 06002.01.321
Book No. 1 TITLE Surface SEM

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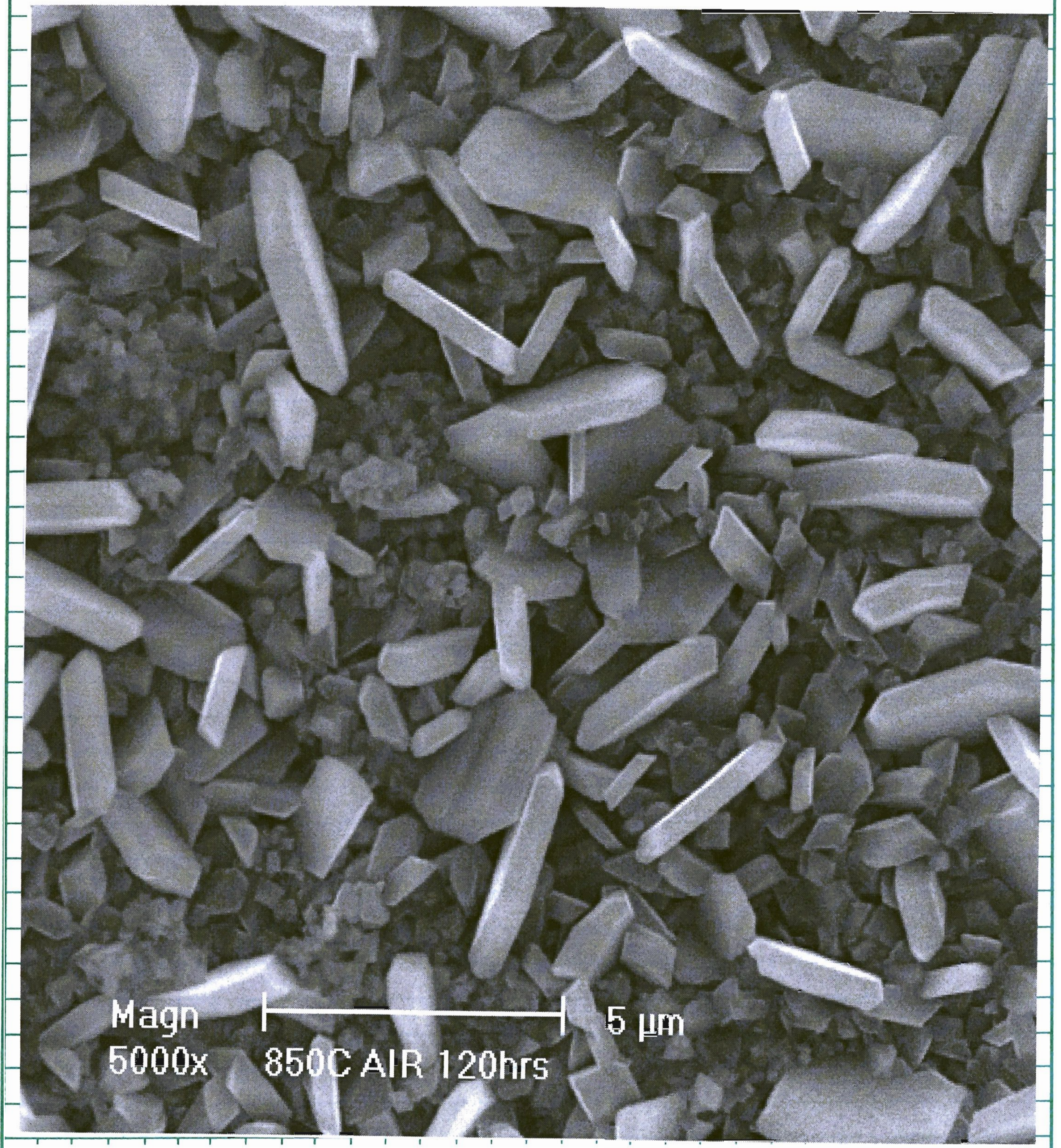


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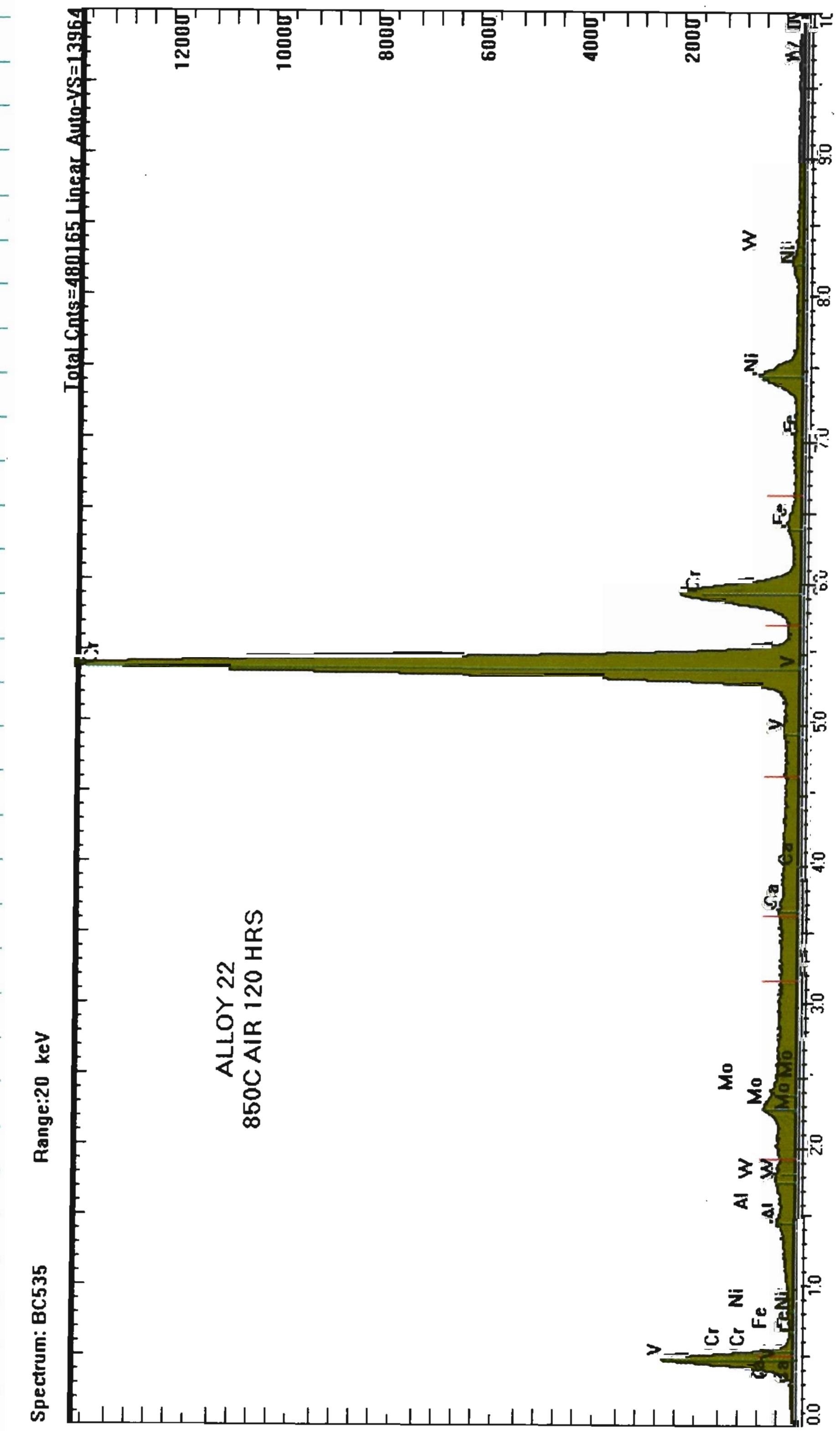
TITLE EDX

Project No. 06002.01.321
Book No. 1



From Page No.

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|---|---|---|---|
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Project No. 06002.01.321
Book No. 1 TITLE EDX

From Page No.

bc535
[A N A L Y S I S   R E P O R T]

```
GENERAL CONDITIONS
------------------
  Result File        : BC535
  File Version       : 1
  Background Method  : Fit
  Decon Method       : Gaussian
  Decon ChiSquared   : 4.03
  Analysis Date      : 14-OCT-2004
  Microscope         : SEM
  Comments           :ALLOY 22 850C AIR 120HRS

ANALYSIS CONDITIONS
-------------------
  Quant. Method        : ZAF/ASAP
  Acquire Time         : 300 secs
  Normalization Factor: 100.00

SAMPLE CONDITIONS
-----------------
  kV                     : 20.0
  Beam Current           : 150.0 picoAmps
  Working Distance       : 29.5 mm
  Tilt Angle             : 0.0 Degrees
  TakeOff Angle          : 43.3 Degrees
  Solid Angle*BeamCurrent: 0.7
```

| Element | Line | Weight% | K-Ratio | Cnts/s | Atomic% |
|---|---|---|---|---|---|
| Al | Ka | 0.40 | 0.0019 | 3.27 | 0.80 |
| Ca | Ka | 0.09 | 0.0010 | 1.11 | 0.12 |
| V | Ka | 0.29 | 0.0029 | 2.47 | 0.31 |
| Cr | Ka | 85.61 | 0.8565 | 640.71 | 87.78 |
| Fe | Ka | 1.28 | 0.0113 | 6.52 | 1.22 |
| Ni | Ka | 9.02 | 0.0851 | 36.06 | 8.19 |
| Mo | La | 2.36 | 0.0195 | 11.07 | 1.31 |
| W | La | 0.94 | 0.0072 | 0.74 | 0.27 |
| Total | | 99.99 | | | |

To Page No.

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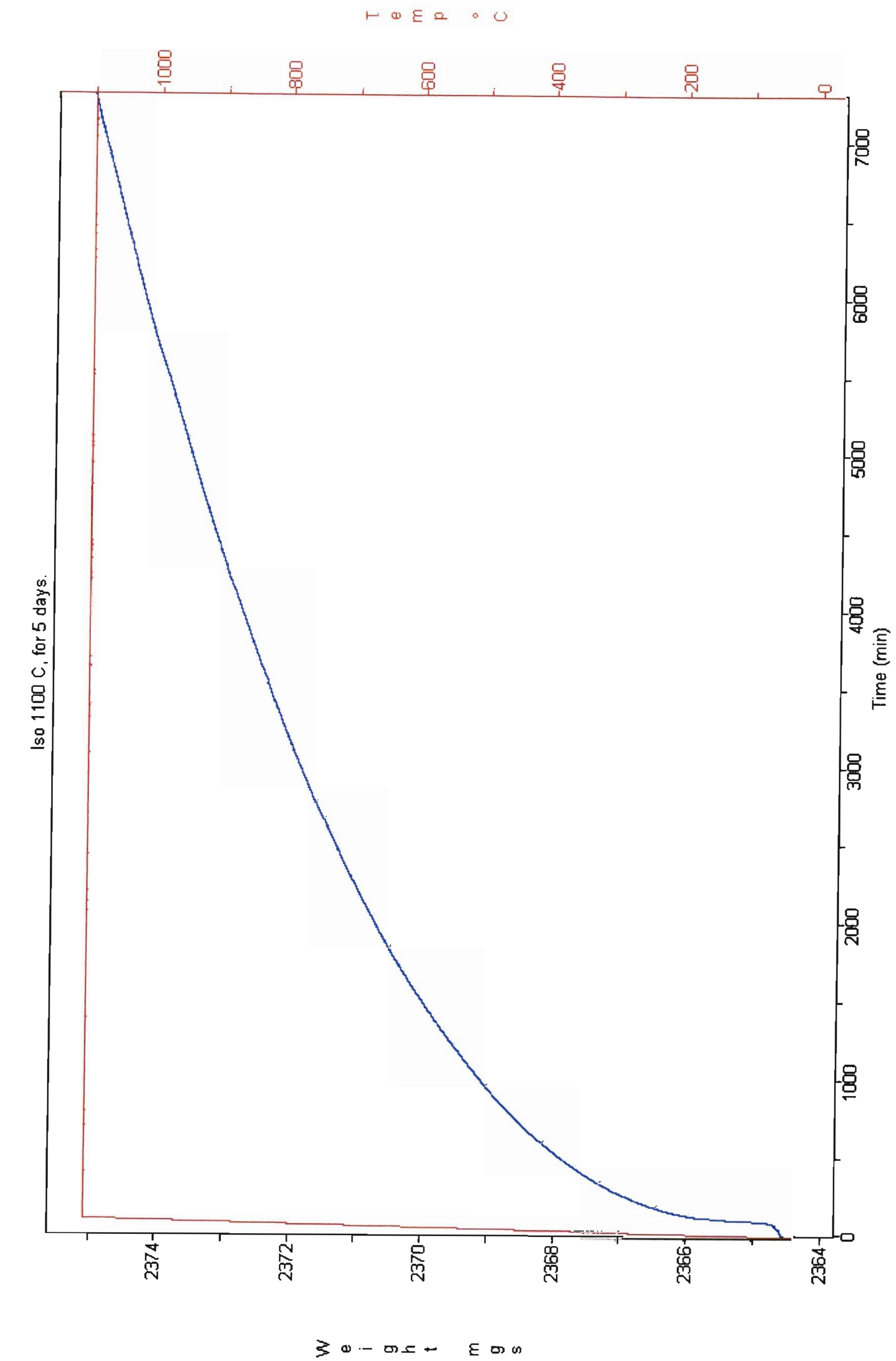
TITLE TGA
Project No. 06002.01.321
Book No. 1

From Page No.

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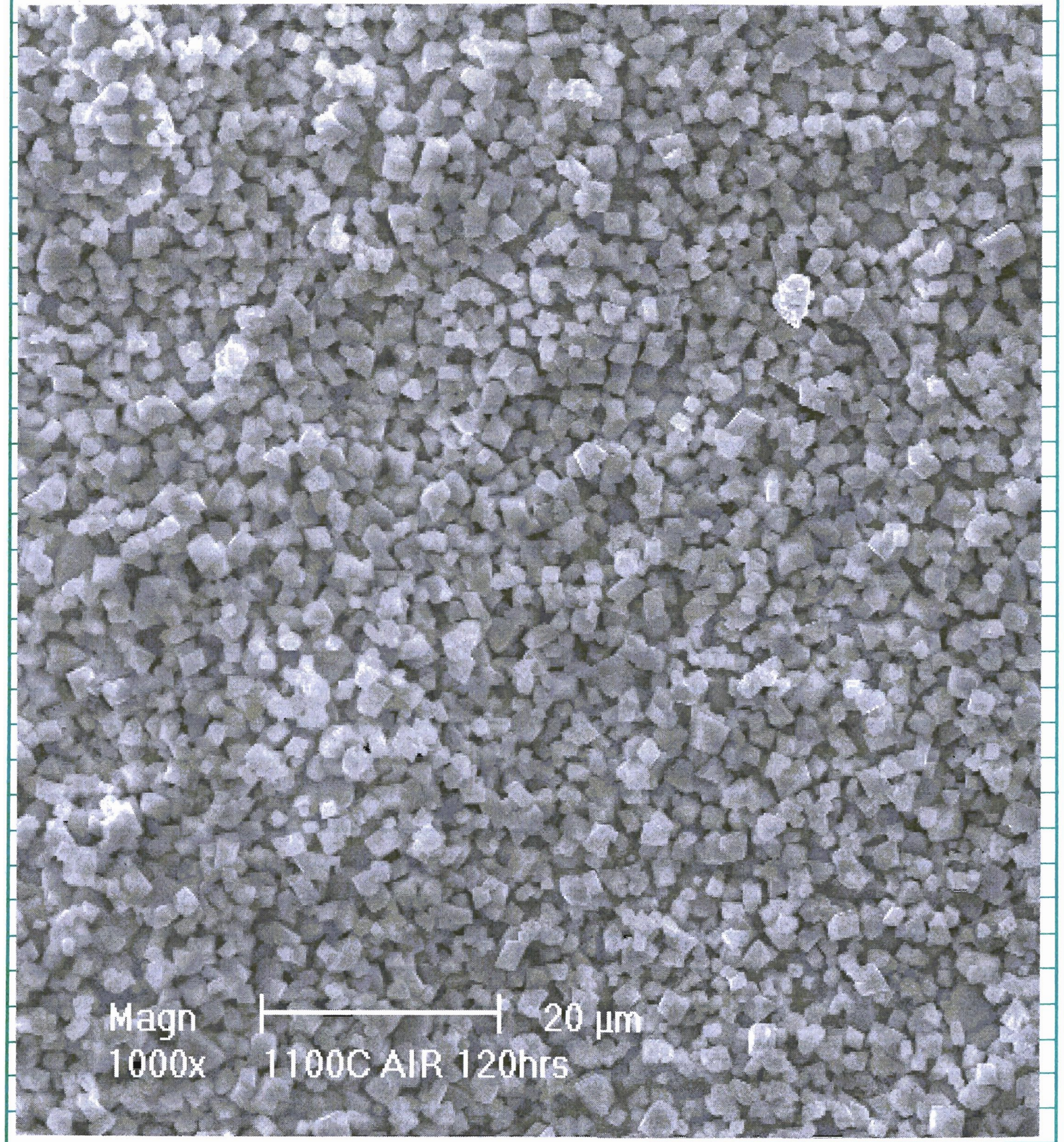
Project No. 06002.01.321
Book No. 1 TITLE Surface SEM

From Page No.

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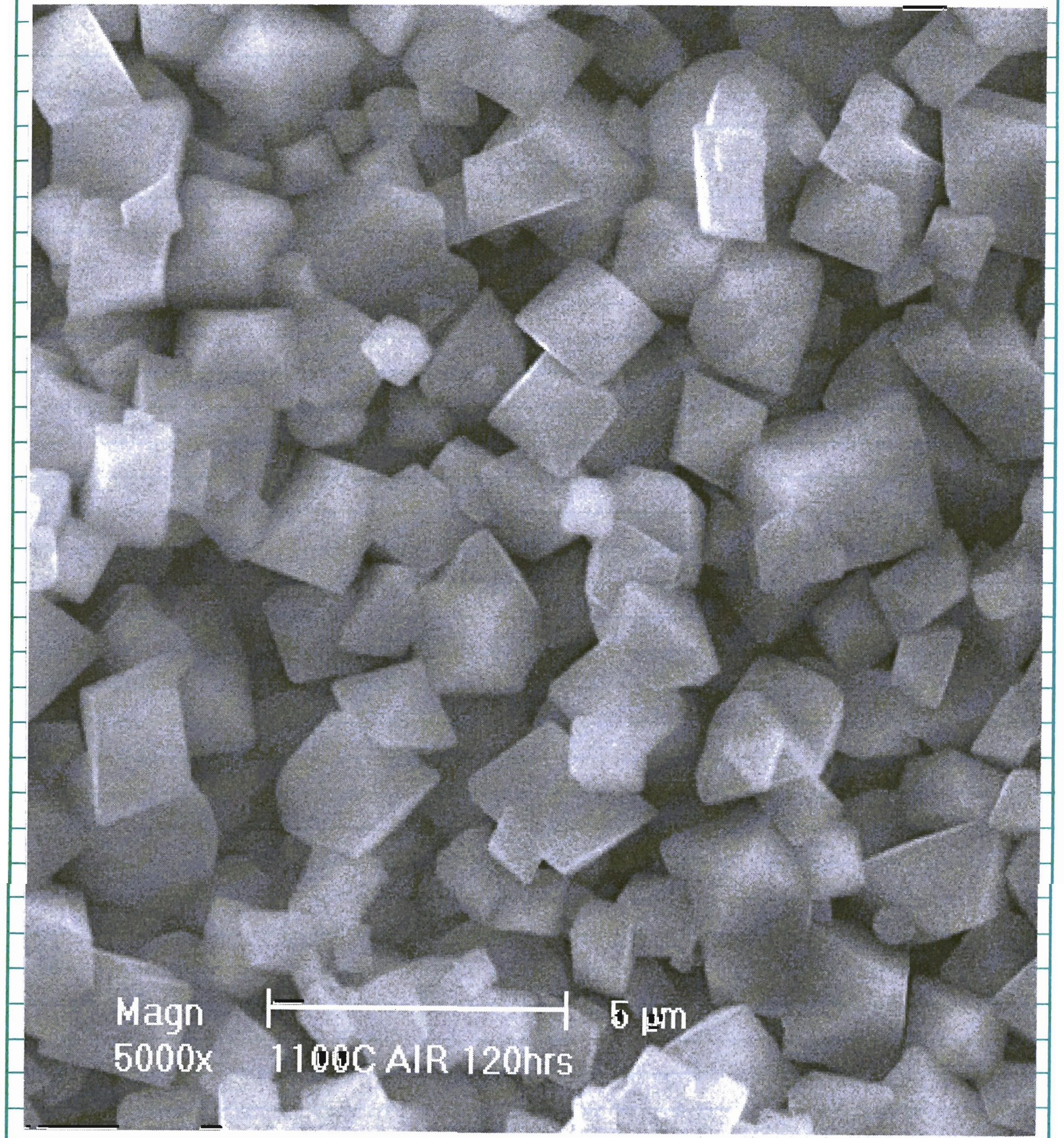
TITLE Surface SEM
Project No. 06002.01.321
Book No. 1



From Page No.

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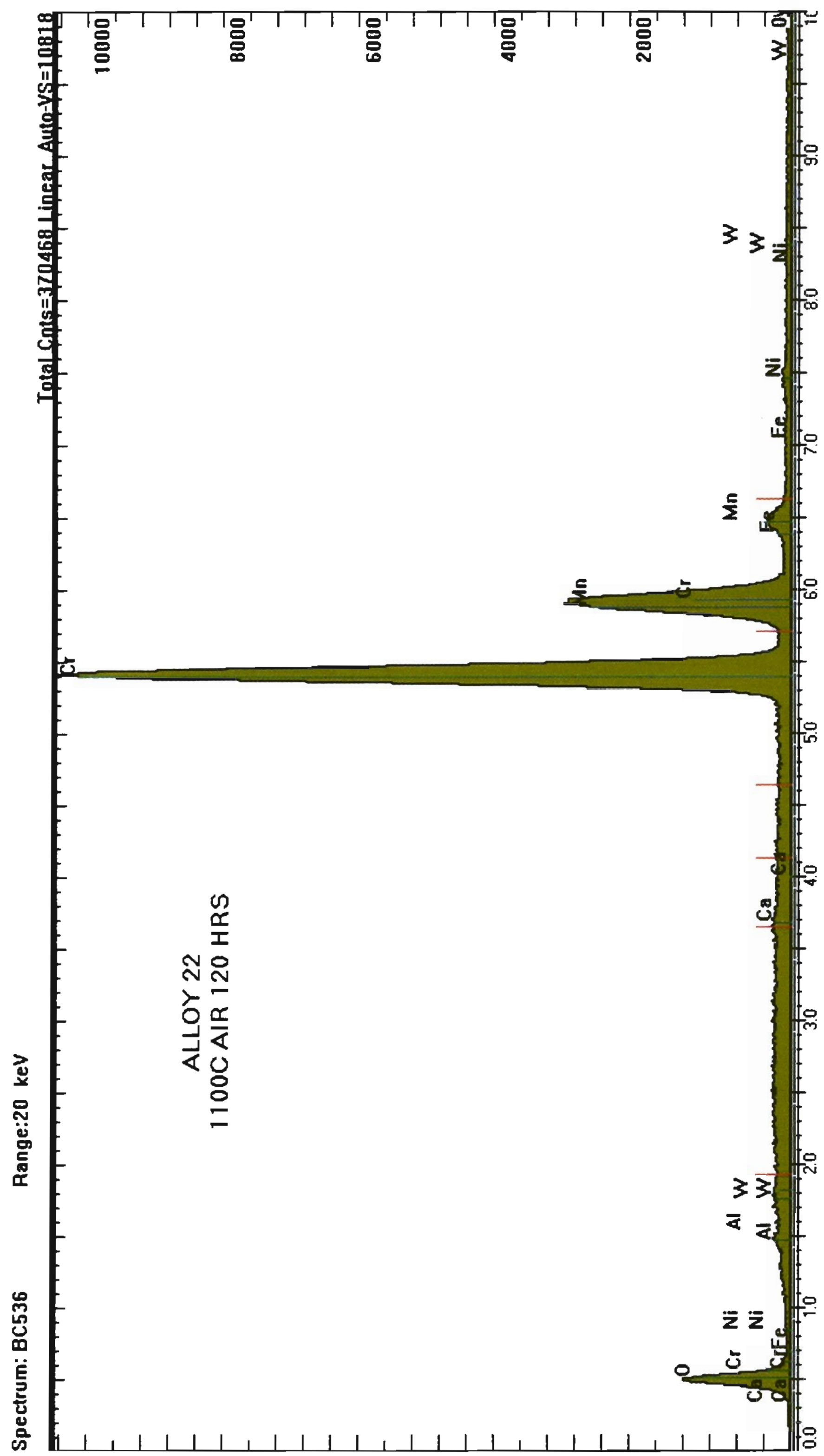
From Page No.

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From Page No.

bc536
[A N A L Y S I S R E P O R T]

GENERAL CONDITIONS

| | |
| --- | --- |
| Result File | : BC536 |
| File Version | : 1 |
| Background Method | : Fit |
| Decon Method | : Gaussian |
| Decon ChiSquared | : 1.77 |
| Analysis Date | : 14-OCT-2004 |
| Microscope | : SEM |
| Comments | :ALLOY 22 1100C AIR 120HRS |

ANALYSIS CONDITIONS

| | |
| --- | --- |
| Quant. Method | : ZAF/ASAP |
| Acquire Time | : 300 secs |
| Normalization Factor | : 100.00 |

SAMPLE CONDITIONS

| | |
| --- | --- |
| kV | : 20.0 |
| Beam Current | : 150.0 picoAmps |
| Working Distance | : 29.5 mm |
| Tilt Angle | : 0.0 Degrees |
| TakeOff Angle | : 43.3 Degrees |
| Solid Angle*BeamCurrent | : 0.7 |

| Element | Line | Weight% | K-Ratio | Cnts/s | Atomic% |
| --- | --- | --- | --- | --- | --- |
| Al | Ka | 0.37 | 0.0018 | 2.45 | 0.72 |
| Ca | Ka | 0.08 | 0.0009 | 0.83 | 0.11 |
| Cr | Ka | 79.49 | 0.7986 | 484.56 | 80.10 |
| Mn | Ka | 19.19 | 0.1899 | 101.66 | 18.30 |
| Fe | Ka | 0.00 | 0.0000 | 0.00 | 0.00 |
| Ni | Ka | 0.88 | 0.0082 | 2.81 | 0.78 |
| W | La | 0.00 | 0.0000 | 0.00 | 0.00 |
| Total | | 100.01 | | | |

To Page No.

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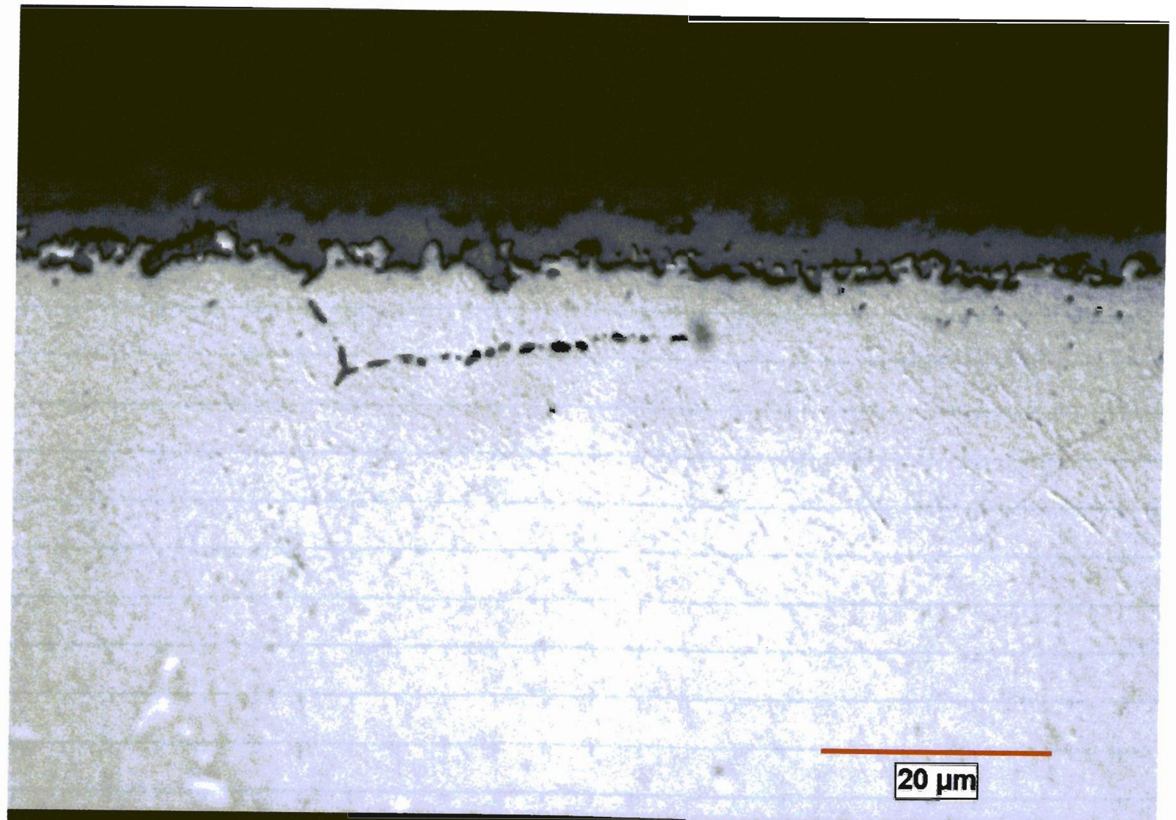
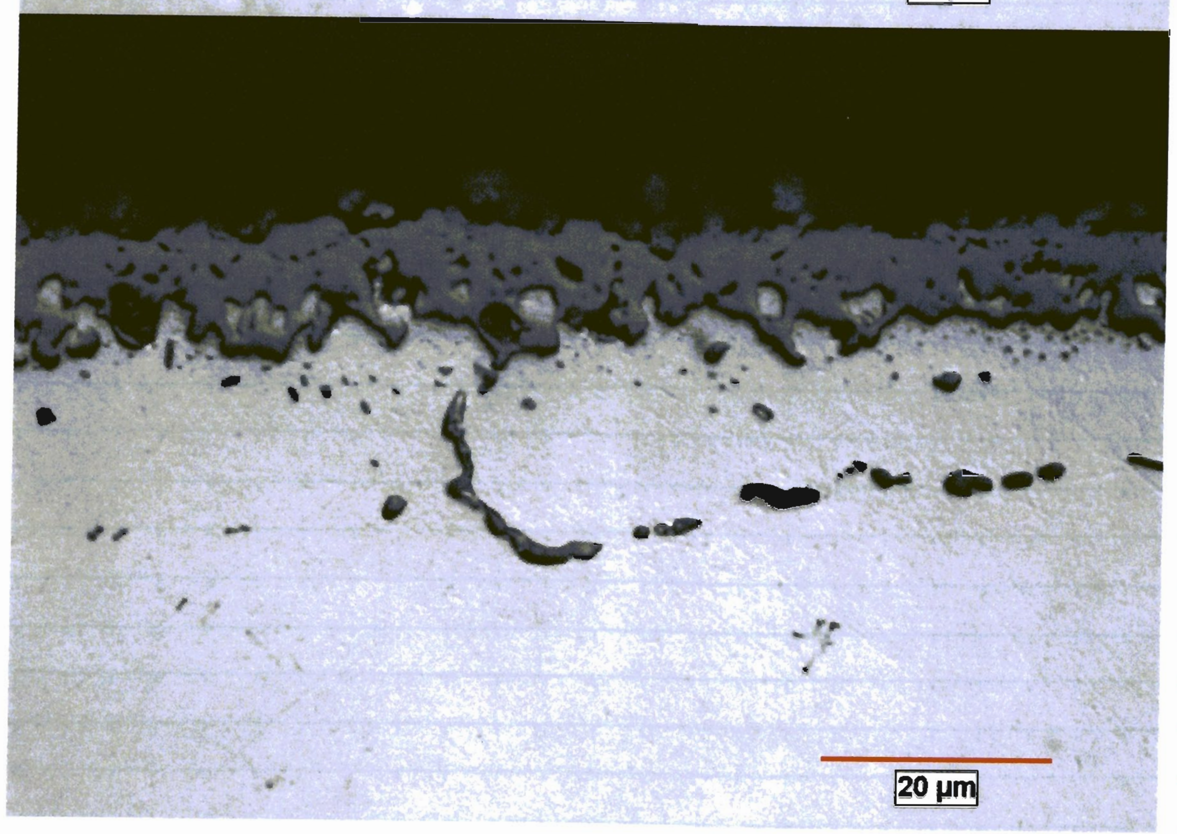
Project No. 06002.01.321
Book No. 1
TITLE Oxide Morphology - Internal Oxidation

From Page No.




1100°C
20h

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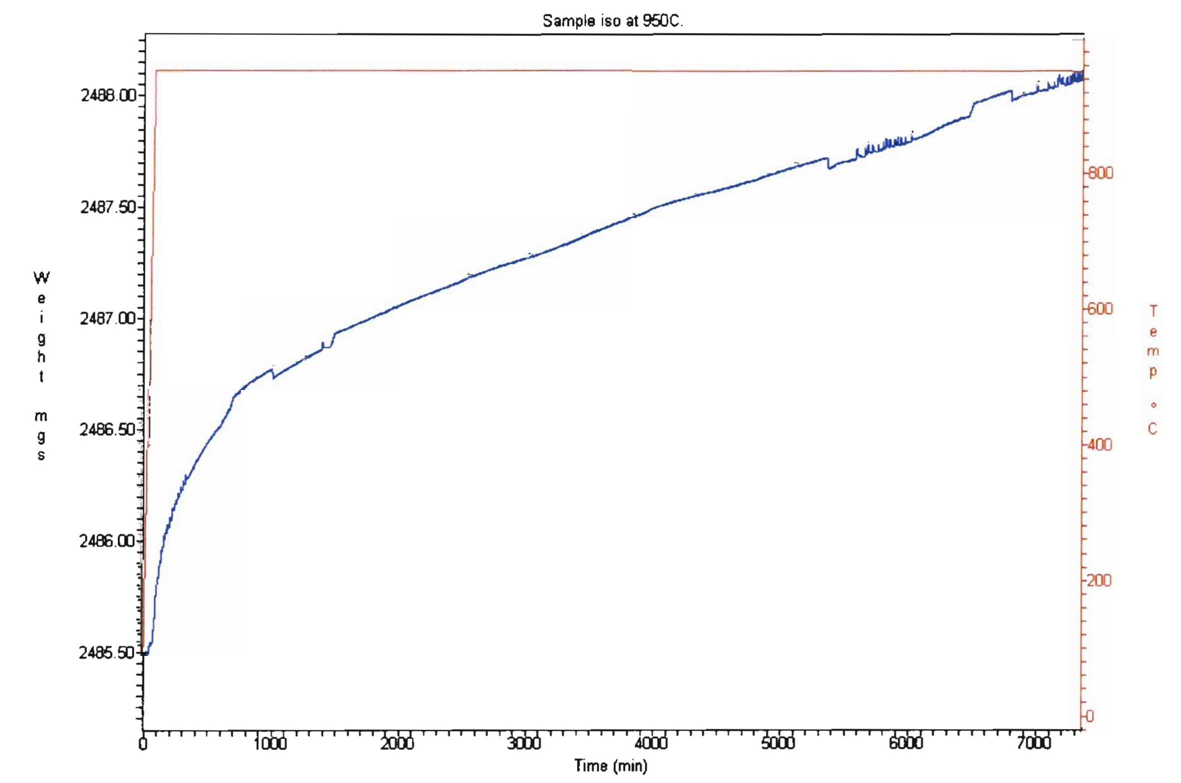
Project No. 06002.01.321
Book No. 1
TITLE TGA Results

From Page No.

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Project No. 06002.01.321
Book No. 1 TITLE TGA Results

From Page No.

| | Time (h) | Time (s) | Wt. (mg) | Wt. Chg (mg) | Wt Chg/area(mg/cm2) |
|---|---|---|---|---|---|
| 1100C | | | | | |
| A=4.51 cm2 | 0 | 0 | 2364.38 | 0 | 0 |
| | 0.1 | 360 | 2364.51 | 0.13 | 0.028824834 |
| | 0.2 | 720 | 2364.53 | 0.15 | 0.033259424 |
| | 0.3 | 1080 | 2364.55 | 0.17 | 0.037694013 |
| | 0.4 | 1440 | 2364.56 | 0.18 | 0.039911308 |
| | 0.6 | 2160 | 2364.57 | 0.19 | 0.042128603 |
| | 0.8 | 2880 | 2364.58 | 0.2 | 0.044345898 |
| | 1 | 3600 | 2364.6 | 0.22 | 0.048780488 |
| 1100C | 1.5 | 5400 | 2364.69 | 0.31 | 0.068736142 |
| | 2 | 7200 | 2365.58 | 1.2 | 0.266075388 |
| | 3 | 10800 | 2366.37 | 1.99 | 0.441241685 |
| | 4 | 14400 | 2366.77 | 2.39 | 0.529933481 |
| | 6 | 21600 | 2367.34 | 2.96 | 0.65631929 |
| | 8 | 28800 | 2367.78 | 3.4 | 0.753880266 |
| | 10 | 36000 | 2368.14 | 3.76 | 0.833702882 |
| | 15 | 54000 | 2368.87 | 4.49 | 0.99556541 |
| | 20 | 72000 | 2369.46 | 5.08 | 1.126385809 |
| | 30 | 108000 | 2370.42 | 6.04 | 1.33924612 |
| | 40 | 144000 | 2371.19 | 6.81 | 1.509977827 |
| | 50 | 180000 | 2371.85 | 7.47 | 1.65631929 |
| | 60 | 216000 | 2372.4 | 8.02 | 1.77827051 |
| | 70 | 252000 | 2372.94 | 8.56 | 1.898004435 |
| | 80 | 288000 | 2373.4 | 9.02 | 2 |
| | 90 | 324000 | 2373.82 | 9.44 | 2.093126386 |
| | 100 | 360000 | 2374.26 | 9.88 | 2.190687361 |
| | 110 | 396000 | 2374.63 | 10.25 | 2.272727273 |
| | 120 | 432000 | 2375 | 10.62 | 2.354767184 |
| | 121.8 | 438480 | 2375.06 | 10.68 | 2.368070953 |

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TITLE TGA Results Book No. 1

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| | Time (h) | Time (s) | Wt. (mg) | Wt. Chg (mg) | Wt Chg/area(mg/cm2) |
|---|---|---|---|---|---|
| 950C | | | | | |
| A=4.532 cr | 0 | 0 | 2485.3 | 0 | 0 |
| | 0.1 | 360 | 2485.45 | 0.15 | 0.03309797 |
| | 0.2 | 720 | 2485.51 | 0.21 | 0.046337158 |
| | 0.3 | 1080 | 2485.5 | 0.2 | 0.044130627 |
| | 0.4 | 1440 | 2485.5 | 0.2 | 0.044130627 |
| | 0.6 | 2160 | 2485.52 | 0.22 | 0.048543689 |
| | 0.8 | 2880 | 2485.51 | 0.21 | 0.046337158 |
| | 1 | 3600 | 2485.52 | 0.22 | 0.048543689 |
| 950C | 1.5 | 5400 | 2485.56 | 0.26 | 0.057369815 |
| | 2 | 7200 | 2485.62 | 0.32 | 0.070609003 |
| | 3 | 10800 | 2486.04 | 0.74 | 0.163283319 |
| | 4 | 14400 | 2486.16 | 0.86 | 0.189761695 |
| | 6 | 21600 | 2486.3 | 1 | 0.220653133 |
| | 8 | 28800 | 2486.43 | 1.13 | 0.249338041 |
| | 10 | 36000 | 2486.52 | 1.22 | 0.269196823 |
| | 15 | 54000 | 2486.75 | 1.45 | 0.319947043 |
| | 20 | 72000 | 2486.81 | 1.51 | 0.333186231 |
| | 30 | 108000 | 2487.02 | 1.72 | 0.379523389 |
| | 40 | 144000 | 2487.16 | 1.86 | 0.410414828 |
| | 50 | 180000 | 2487.29 | 1.99 | 0.439099735 |
| | 60 | 216000 | 2487.42 | 2.12 | 0.467784643 |
| | 70 | 252000 | 2487.55 | 2.25 | 0.49646955 |
| | 80 | 288000 | 2487.64 | 2.34 | 0.516328332 |
| | 90 | 324000 | 2487.7 | 2.4 | 0.52956752 |
| | 100 | 360000 | 2487.81 | 2.51 | 0.553839365 |
| | 110 | 396000 | 2488 | 2.7 | 0.59576346 |
| | 120 | 432000 | 2488.07 | 2.77 | 0.611209179 |
| | 122.5 | 441160 | 2488.12 | 2.82 | 0.622241836 |

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Project No 06002.01.321
Book No. 1 TITLE TGA Results

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| | | | | | |
|---|---|---|---|---|---|
| 850C | Time (h) | Time (s) | Wt. (mg) | Wt. Chg (mg) | Wt Chg/area(mg/cm2) |
| A=4.534 cr | 0 | 0 | 2350.83 | 0 | 0 |
| | 0.1 | 360 | 2350.95 | 0.12 | 0.026466696 |
| | 0.2 | 720 | 2350.99 | 0.16 | 0.035288928 |
| | 0.3 | 1080 | 2351.01 | 0.18 | 0.039700044 |
| | 0.4 | 1440 | 2351.02 | 0.19 | 0.041905602 |
| | 0.6 | 2160 | 2351.03 | 0.2 | 0.04411116 |
| | 0.8 | 2880 | 2351.05 | 0.22 | 0.048522276 |
| | 1 | 3600 | 2351.07 | 0.24 | 0.052933392 |
| 950C | 1.5 | 5400 | 2351.15 | 0.32 | 0.070577856 |
| | 2 | 7200 | 2351.19 | 0.36 | 0.079400088 |
| | 3 | 10800 | 2351.24 | 0.41 | 0.090427878 |
| | 4 | 14400 | 2351.27 | 0.44 | 0.097044552 |
| | 6 | 21600 | 2351.33 | 0.5 | 0.1102779 |
| | 8 | 28800 | 2351.38 | 0.55 | 0.12130569 |
| | 10 | 36000 | 2351.41 | 0.58 | 0.127922364 |
| | 15 | 54000 | 2351.49 | 0.66 | 0.145566828 |
| | 20 | 72000 | 2351.56 | 0.73 | 0.161005734 |
| | 30 | 108000 | 2351.65 | 0.82 | 0.180855757 |
| | 40 | 144000 | 2351.74 | 0.91 | 0.200705779 |
| | 50 | 180000 | 2351.83 | 1 | 0.220555801 |
| | 60 | 216000 | 2351.89 | 1.06 | 0.233789149 |
| | 70 | 252000 | 2351.97 | 1.14 | 0.251433613 |
| | 80 | 288000 | 2352.02 | 1.19 | 0.262461403 |
| | 90 | 324000 | 2352.06 | 1.23 | 0.271283635 |
| | 100 | 360000 | 2352.1 | 1.27 | 0.280105867 |
| | 110 | 396000 | 2352.14 | 1.31 | 0.288928099 |
| | 120 | 432000 | 2352.19 | 1.36 | 0.299955889 |
| | 121.4 | 436970 | 2352.19 | 1.36 | 0.299955889 |

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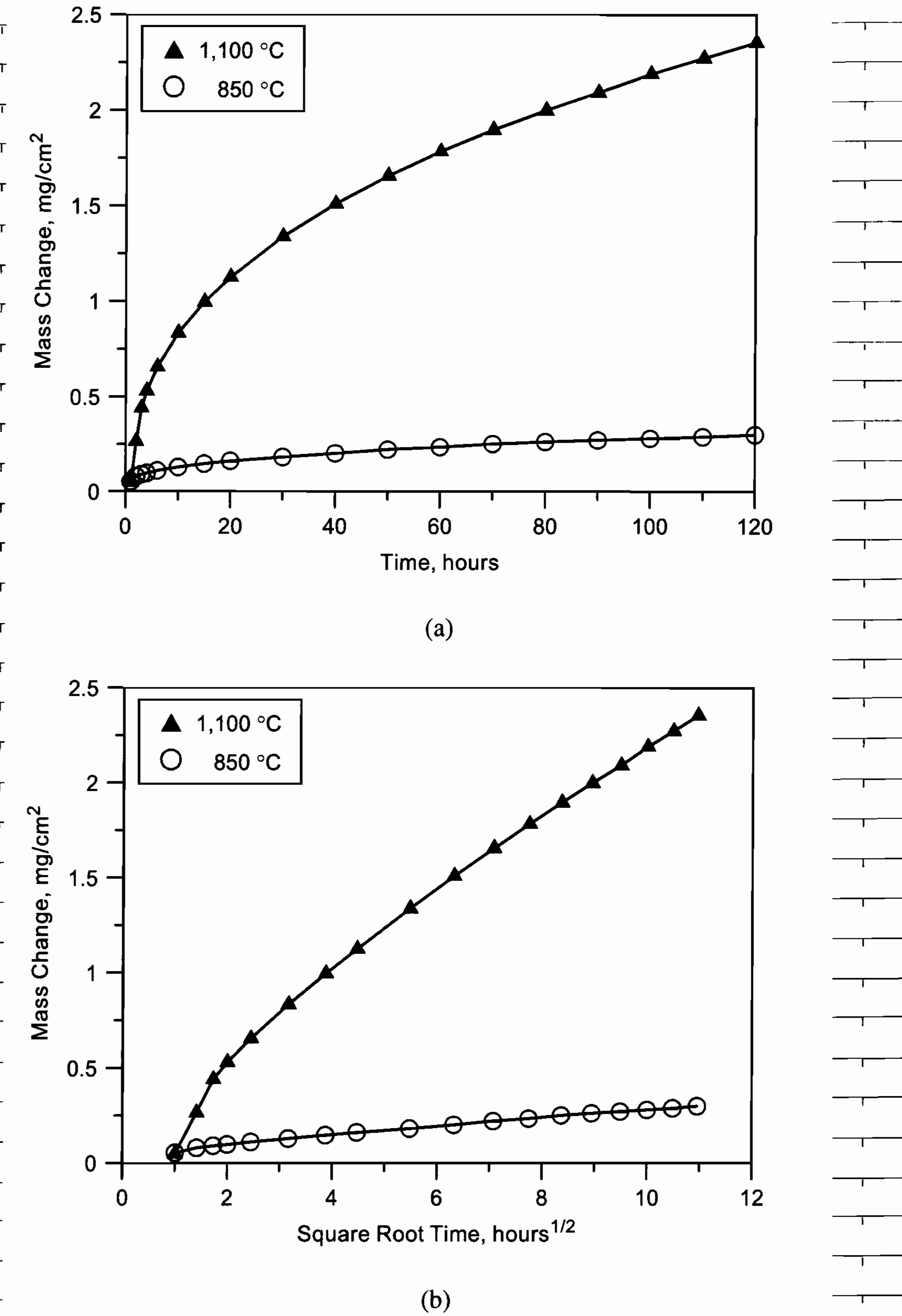
TITLE Oxidation Kinetics
Project No. 06002.01.322
Book No. 1

From Page No.

Oxidation Kinetics of Alloy 22 in Air at 850 °C [1,562 °F] and 1,100 °C [2,012 °F]: (a) Mass Change Versus Time Data; (b) Mass Change Because of Oxidation . . .

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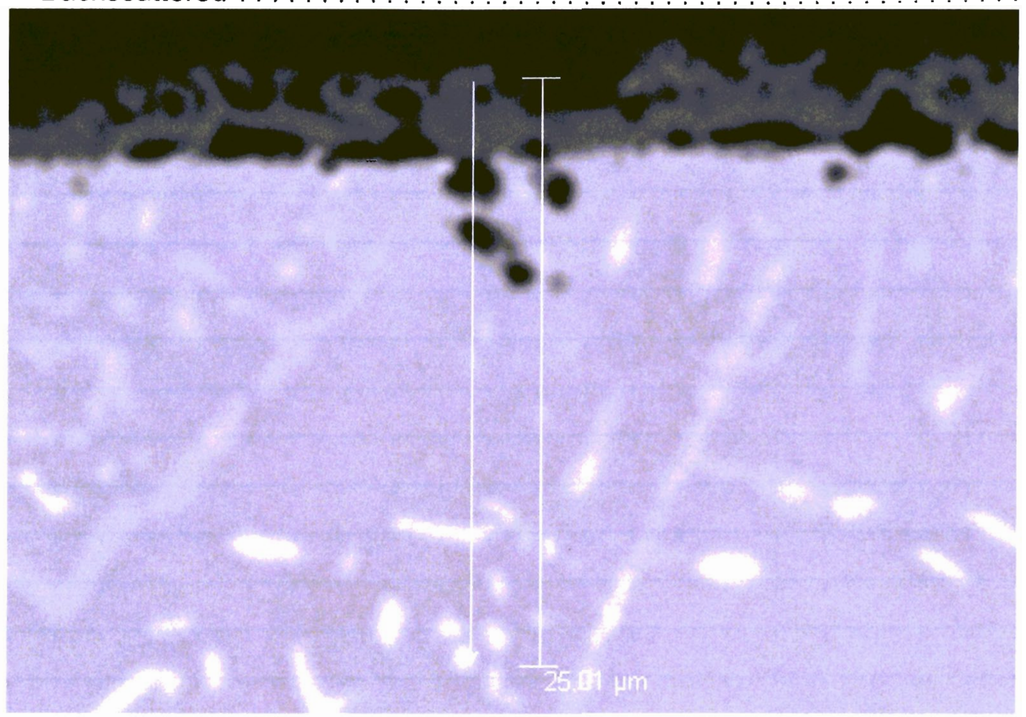
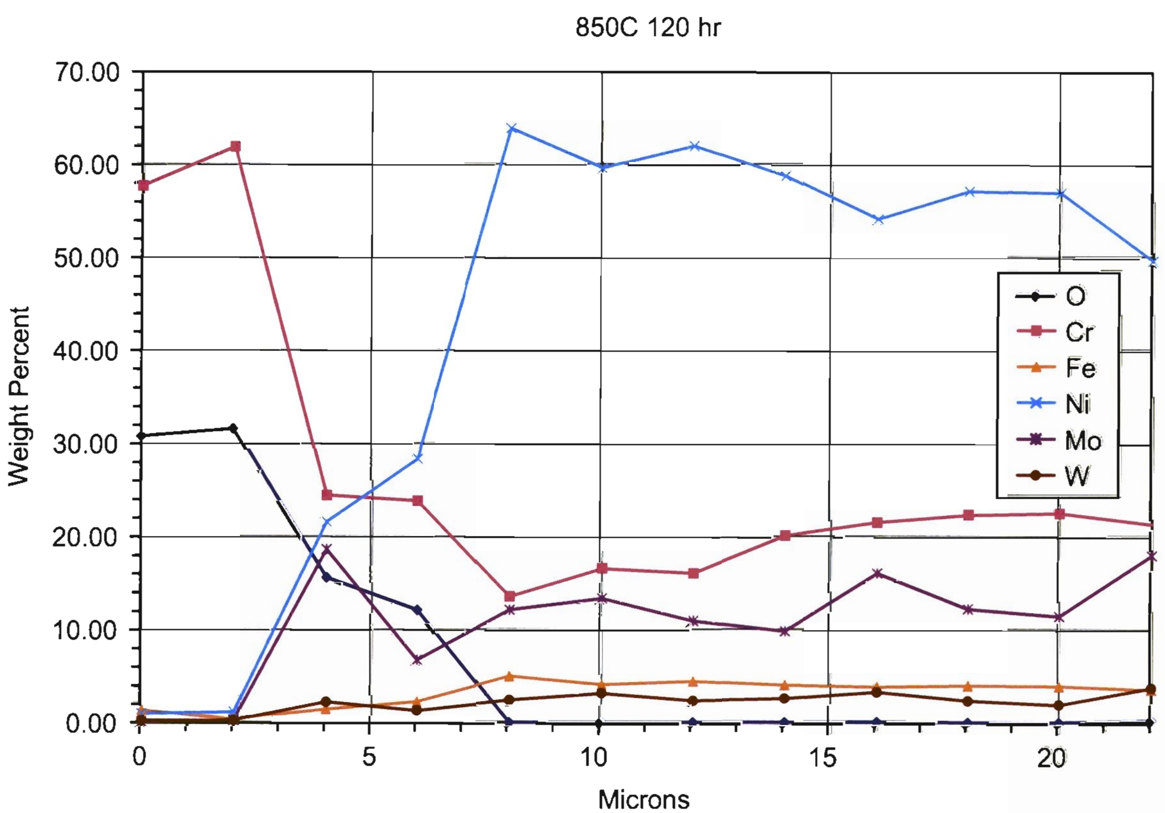
From Page No.

Cross-Section Through the Scale Produced on Alloy 22 After 120 Hours Oxidation at 850 °C [1,562 °F]: (a) Scanning Electron Microscope Backscattered




(a)



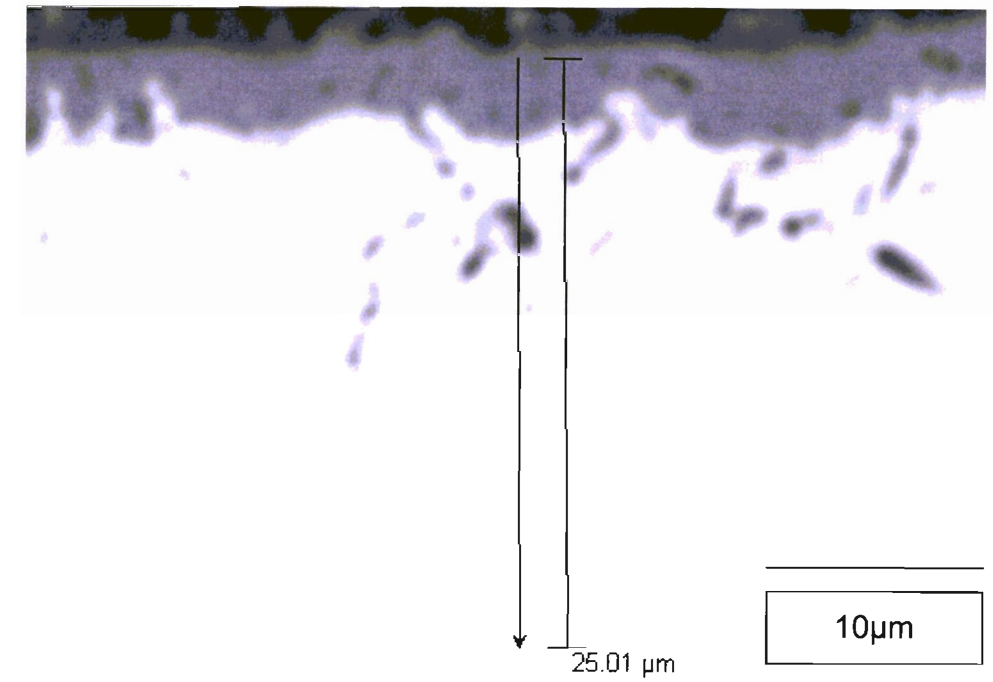

(b)



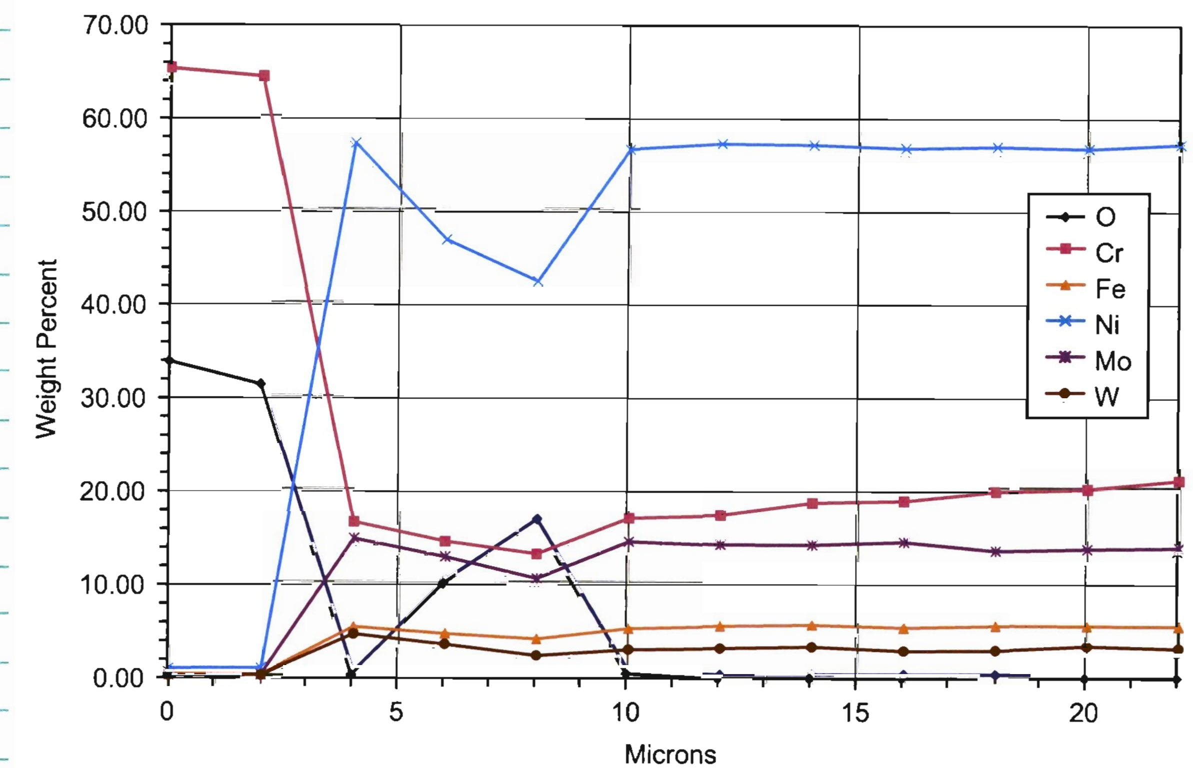


From Page No.

Cross-Section Through the Scale Produced on Alloy 22 After 24 Hours Oxidation at 1,100 °C [2,012 °F]: (a) Scanning Electron Microscope Backscattered



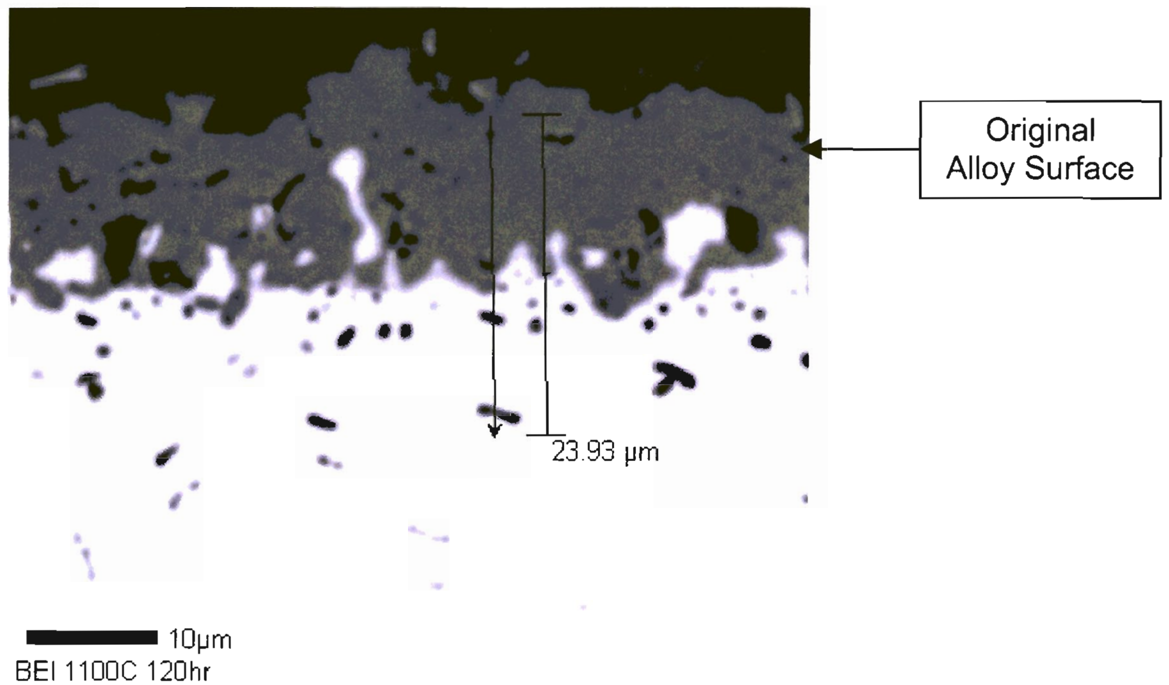

(a)



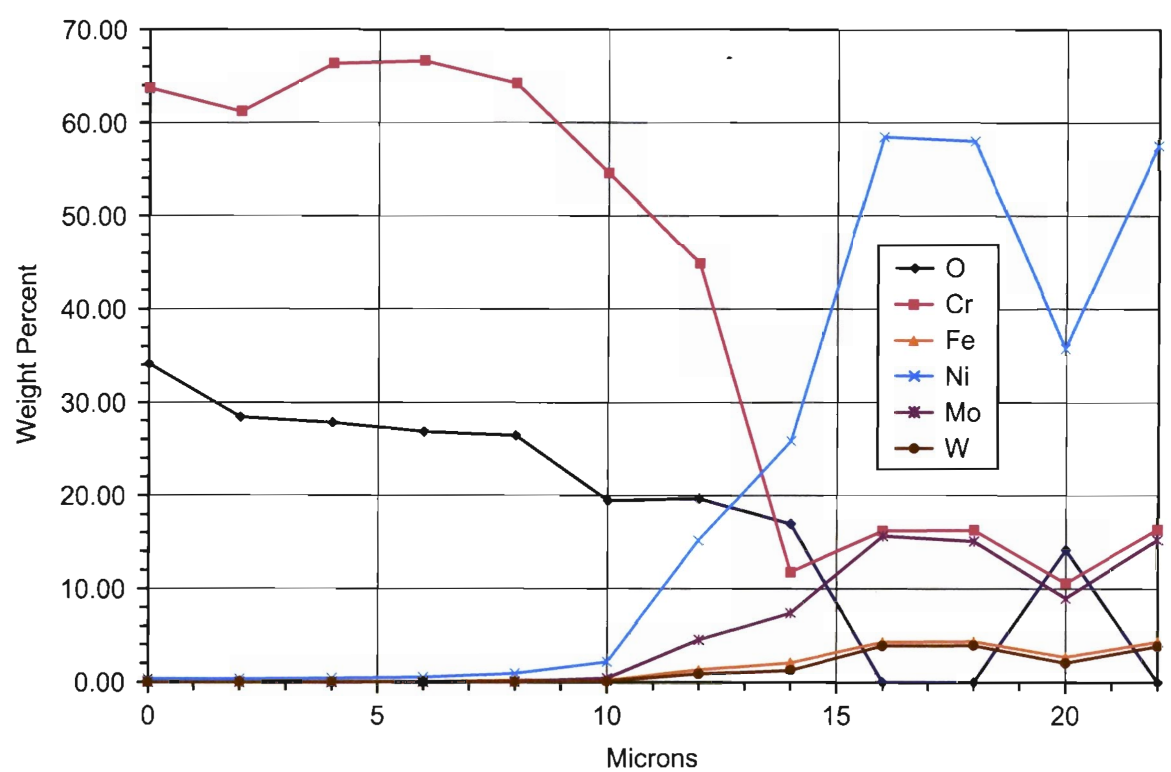

(b)

From Page No.

Oxide Morphology for Alloy 22 Oxidized in Air for 120 Hours at 1,100 °C [2,012 °F]: (a) Scanning Electron Microscope Backscattered Electron Image . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . .




(a)




(b)

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|---|---|---|---|
| | | Recorded by K. T. Chiang | 2/25/05 |



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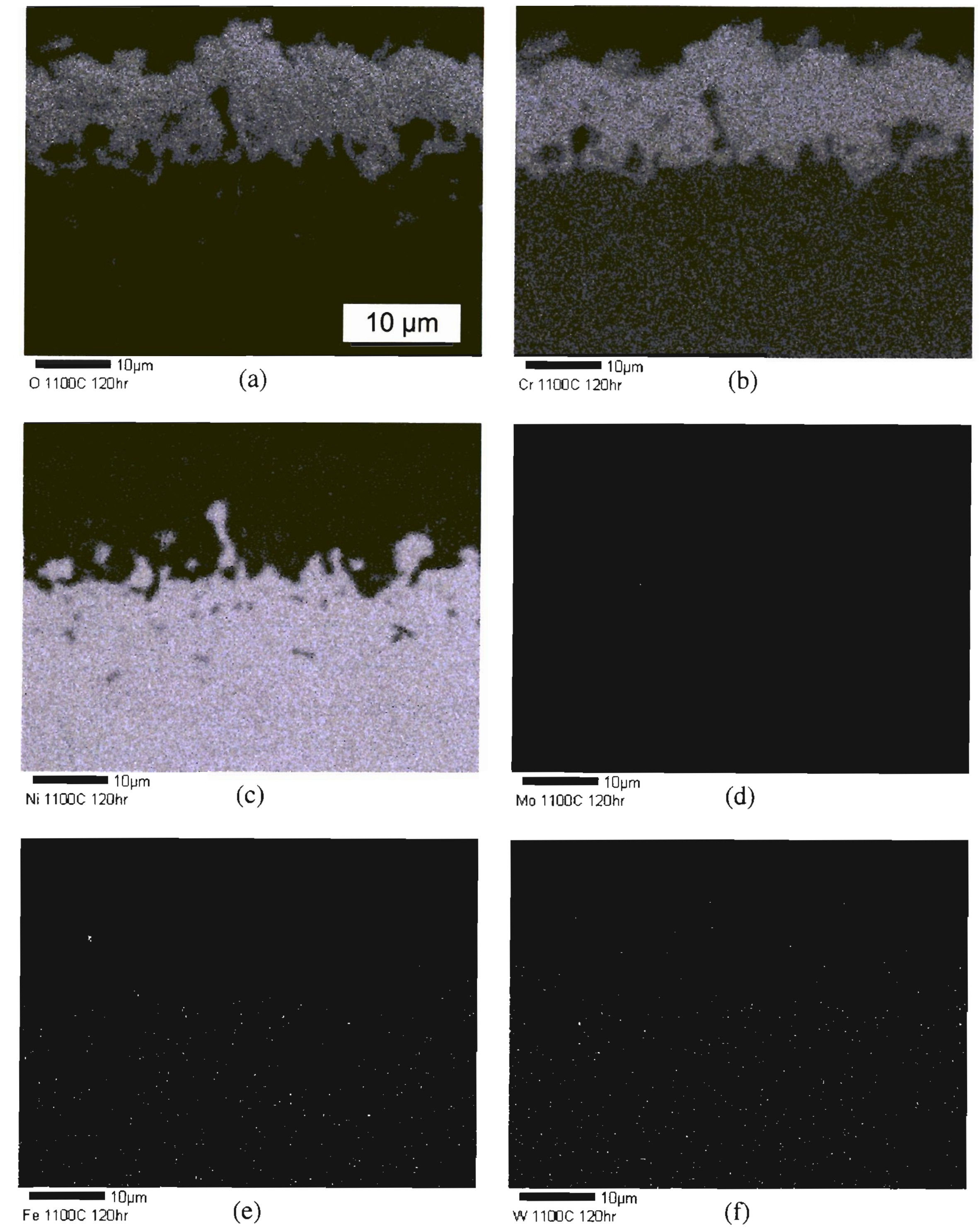
Distribution of Oxygen, Chromium, Iron, Molybdenum, and Tungsten in the Area Outlined in Figure 2-4a: (a) Oxygen X-Ray Map; (b) Chromium X-Ray Map . . . . . .




(a) (b) (c) (d) (e) (f)

To Page No.

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|---|---|---|---|
| | | Recorded by K. T. Chiang | 2/25/05 |

From Page No.

## 2 PASSIVE AND LOCALIZED CORROSION OF OVERPACK MATERIALS—MODELING AND EXPERIMENTS

### 2.1 Dry-Air Oxidation

Dry-air oxidation is assumed to occur during the initial period after waste package emplacement when the radioactive decay heat keeps moisture away from the emplacement drifts (Bechtel SAIC Company, LLC, 2003a). According to the DOE model (Bechtel SAIC Company, LLC, 2003a; Welsch, et al., 1996), dry-air oxidation of Alloy 22 results in the formation of an adherent, protective $Cr_2O_3$ film of uniform thickness. The $Cr_2O_3$ film growth rate, oxidation time, and temperature relationship are described in Eqs. (2-1) through (2-3). The $Cr_2O_3$ film growth rate follows a parabolic rate law, where the film thickness or mass gain is proportional to the square root of time

$$\Delta m/A = (K_p t)^{1/2} + C \qquad (2\text{-}1)$$

where

$\Delta m/A$ — mass gain per unit area in time $t$ (mg cm$^{-2}$)
$K_p$ — parabolic rate constant (mg$^2$cm$^{-4}$h$^{-1}$)
$C$ — a constant (mg cm$^{-2}$)

The temperature dependence of the oxidation rate is contained in the parabolic rate constant

$$K_p = K_o \exp(-Q_{oxid}/RT) \qquad (2\text{-}2)$$

where

$K_o$ — a constant (mg$^2$cm$^{-4}$h$^{-1}$)
$Q_{oxid}$ — activation energy for oxide growth (J mol$^{-1}$)
$R$ — the ideal gas constant (J mol$^{-1}$ K$^{-1}$)
$T$ — absolute temperature (K)

Taking the logarithm on both sides of Eq. (2-2), the equation can be rewritten as

$$\text{Log } K_p = \text{Log } K_o - Q_{oxid}/RT \qquad (2\text{-}3)$$

The activation energy for oxide growth $Q_{oxid}$ can be determined experimentally by performing oxidation tests at elevated temperatures. The temperature dependence of $K_p$ is Arrhenius type. For the Arrhenius temperature relationship, the oxidation rate of Alloy 22 for long time exposures at lower temperatures can be extrapolated from short-term tests at higher temperatures.

In this investigation the oxidation kinetics and oxide morphology of Alloy 22 in dry-air were studied at elevated temperatures of 850 °C [1,562 °F] and 1,100 °C [2,012 °F] and extrapolated to expected repository temperatures. Special attention was paid to the phases of oxides that formed, as well as the extent of outward diffusion of chromium and inward penetration of oxygen. The results are analyzed and the potential effect of dry-air oxidation on waste package life is discussed.



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The purpose of this work was to identify if there are correlations that allow oxide growth rate and depth of oxygen penetration to be estimated as a function of time and temperature. These tests enhance fundamental understanding of oxide film properties during the thermal pulse period and, on subsequent corrosion in an aqueous environment.

### 2.2 Experimental

The composition of the alloys used in this study is shown in Table 2-1. The oxidation test specimens were machined from mill-annealed, 12.7-mm [0.5-in]-thick plate stock. The plate material was confirmed to meet the chemical composition specified in ASTM B–575 for UNS N06022 alloy designation (ASTM International, 2004a). The machined plate was cut into oxidation coupons of dimension 15 × 13 × 1.5 mm [0.6 × 0.5 × 0.06 in]. Specimens were wet surface ground with 600-grit silicon carbide paper and ultrasonically cleaned in a acetone and alcohol baths before oxidation exposure.

The oxidation tests were performed with a microbalance[1] that enabled continuous monitoring of weight change and temperature. After oxidation, cross sections of the specimens were examined optically and with an electron microprobe[2] and associated wavelength x-ray spectrometers. In this analysis, a backscattered electron image was taken of the surface oxide layer and the internal oxygen penetration region. Quantitative line scans for oxygen, chromium, iron, nickel, molybdenum, and tungsten were then run starting at the surface and penetrating in 2 µm [78.7 µin] increments into the base material. For selected samples, x-ray dot maps of the elements also were made to show the elemental distribution in the oxide/alloy interface region. In this analysis, the detector was tuned to respond to x-ray wavelengths characteristic of oxygen, chromium, nickel, molybdenum, iron, and tungsten. The density of the white dots therefore provided information on the abundance of each element in the examined areas. The intensities in the x-ray maps do not directly correlate quantitatively with element concentrations.

### 2.3 Oxidation Rate Measurements

The oxidation rate data for Alloy 22 specimens subjected to isothermal oxidation in air at 850 °C [1,562 °F] and 1,100 °C [2,012 °F] are presented in Figure 2-1(a) as mass change versus time. The oxidation mass change at a given exposure time is greater at 1,100 °C [2,012 °F] than at 850 °C [1562 °F]. The results are expected because the oxidation rate is higher at higher temperatures. At both temperatures, the alloy exhibited a much more rapid growth rate during an initial transient period of approximately 1.5 hours. The rate slowed at longer times. The mass change data are also plotted as mass change versus the square root of time in Figure 2-1(b). For the 850 °C [1,562 °F] specimens, the data fall along a straight line indicating a parabolic rate law for oxidation kinetics.

The mass gain during oxidation is the result of the external oxide formation and the mass of oxygen absorbed in the alloy. Wagner developed the theory of high temperature oxidation of metals under idealized conditions (Wagner, 1959). The theory describes the oxidation behavior for the case where the diffusion of ions in the metal is rate limiting, the oxidation follows a

[1]Cahn TherMax, 700 Microbalance, Thermo Electron Corporation, Newington, New Hampshire.

[2]JEOL, JXA–8600 Electron Microprobe, JEOL USA, Inc., Peabody, Massachusetts.



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**Table 2-1. Composition of Alloys 22, 625, and 825 Heats and Alloy 622 Filler Metal**

| Material | Ni* | Cr | Mo | W | Fe | Co | Si | Mn | V | P | S | C | Other |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Alloy 22 Heat 2277-8-3175 12.7-mm | 57.8 | 21.40 | 13.60 | 3.00 | 3.80 | 0.09 | 0.030 | 0.12 | 0.15 | 0.008 | 0.002 | 0.004 | — |
| Alloy 22 Heat 2277-8-3235 12.7-mm | Bal | 21.40 | 13.47 | 2.87 | 3.94 | 1.31 | 0.023 | 0.24 | 0.17 | 0.008 | 0.001 | 0.003 | — |
| 622 Filler Heat XX1045BG1 | Bal | 20.73 | 14.13 | 3.15 | 3.05 | 0.09 | 0.060 | 0.24 | 0.01 | 0.007 | 0.001 | 0.006 | — |
| Alloy 22 Heat 059902LL2 38.1-mm | Bal | 20.35 | 13.85 | 2.63 | 2.85 | 0.01 | 0.05 | 0.16 | 0.17 | 0.007 | 0.0002 | 0.005 | — |
| 622 Filler Heat XX2048BG | Bal | 20.48 | 14.21 | 3.02 | 2.53 | 0.02 | 0.07 | 0.20 | 0.02 | 0.009 | <0.001 | 0.001 | — |
| Alloy 22 Heat 2277-3-3266 12.7 mm | Bal | 21.40 | 13.30 | 2.81 | 3.75 | 1.19 | 0.03 | 0.23 | 0.14 | 0.008 | 0.004 | 0.005 | — |
| Alloy 22 Heat 2277-1-3164 25.4 mm | Bal | 21.15 | 13.47 | 3.26 | 3.93 | 1.27 | 0.02 | 0.23 | 0.11 | 0.007 | 0.003 | 0.003 | — |
| ERNiCrMo-10 filler heat WN813 | Bal | 22.24 | 13.7 | 3.13 | 2.37 | 0.41 | 0.02 | 0.34 | 0.01 | 0.003 | 0.001 | 0.003 | — |
| ERNiCrMo-10 filler heat XX1977BG1 | Bal | 20.25 | 14.13 | 2.99 | 2.56 | 0.07 | 0.06 | 0.20 | 0.04 | 0.008 | 0.001 | 0.005 | — |
| Alloy 625 NX9936AG 12.7-mm | 60.9 | 21.70 | 9.01 | — | 3.96 | 0.15 | 0.16 | 0.08 | — | 0.009 | 0.001 | 0.02 | Nb: 3.48 Al: 0.24 Ti: 0.24 |
| Alloy 825 HH4371FG 12.7-mm | 41.06 | 22.09 | 3.21 | — | 30.41 | — | 0.29 | 0.35 | — | — | — | 0.010 | Cu: 1.79 Al: 0.07 Ti: 0.82 |

*Ni—nickel, Cr—chromium, Mo—molybdenum, W—tungsten, Fe—iron, Co—cobalt, Si—silicon, Mn—manganese, V—vanadium, P—phosphorus, S—sulfur, C—carbon



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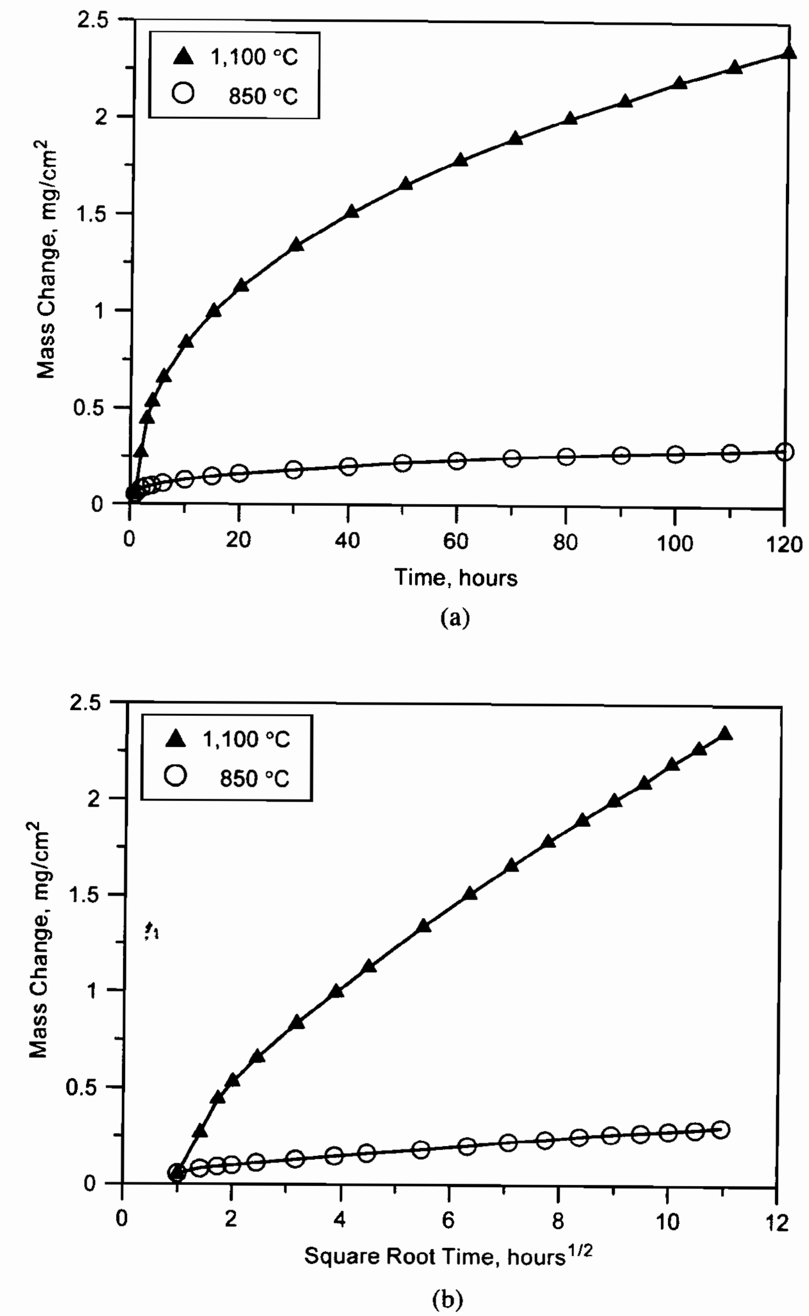


**Figure 2-1. Oxidation Kinetics of Alloy 22 in Air at 850 °C [1,562 °F] and 1,100 °C [2,012 °F]: (a) Mass Change Versus Time Data; (b) Mass Change Because of Oxidation Versus Square Root of Time Data**



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parabolic rate law. The parabolic rate constant [the square of the slope from Figure 2-1(b)] for Alloy 22 at 850 °C [1,562 °F] is 5.1 × $10^{-4}$ $mg^2/cm^4h$. At 1,100 °C [2,012 °F], however, the data for mass gain versus square root of time do not plot as a straight line. The slope of the plot decrease with increasing exposure time, indicating a complex oxidation mechanism.

## 2.4 Oxidation Morphology and Assessment of Intergranular Oxidation

### 2.4.1 Scale Morphology and Composition at 850 °C [1,562 °F]

A cross-section view of the oxides that formed after exposure for 120 hours at 850 °C [1,562 °F] is shown in Figure 2-2(a). The elemental line scans of oxygen, chromium, iron, nickel, molybdenum, and tungsten are presented in Figure 2-2(b). The external scale consisted primarily of chromium and oxygen, indicating the oxide is $Cr_2O_3$. The $Cr_2O_3$ is approximately 4 µm [157.4 µin] thick. In several localized areas, internal oxide precipitates were also observed underneath the external scale. These internal oxides appeared as dark spots in the backscattered electron micrograph in Figure 2-2(a). The maximum depth of inward oxygen penetration is approximately 4 µm [157.4 µin]. The composition profiles also show that there is a chromium depletion zone of about 8 µm [314.9 µin] underneath the external scale. The chromium content in the chromium depletion zone is about 16 wt% [Figure 2-2(b)], which is lower than the 21.4 wt% chromium in the bulk alloy (Table 2-1). There is a chromium concentration gradient underneath the external $Cr_2O_3$ scale which is the driving force for outward chromium diffusion. Nickel is slightly enriched in the chromium depletion zone because of the chromium deficit. No significant diffusion of iron, molybdenum, or tungsten was observed.

### 2.4.2 Scale Morphology and Composition at 1,100 °C [2,012 °F]

A cross-section view of the oxides that formed after exposure for 24 hours at 1,100 °C [2,012 °F] is shown in Figure 2-3(a). The elemental line scans of oxygen, chromium, iron, nickel, molybdenum, and tungsten are presented in Figure 2-3(b). A continuous layer of $Cr_2O_3$ was formed [see concentration profiles of chromium and oxygen near the surface in Figure 2-3(b)]. The thickness of the external scale ranged from 2–4 µm [78.7–157.4 µin]. Oxide precipitates were also observed along grain boundaries deep into the alloy. The chromium concentration around the oxide precipitates is estimated to be 14 wt%. Between 10 to 20 µm [393.7 to 787.4 µin] from the surface, there is a chromium concentration gradient, which is the driving force for outward chromium diffusion. The maximum depth of inward oxygen penetration is about 10 µm [393.7 µin].

The oxides that formed on Alloy 22 after the longer time exposure of 120 hours at 1,100 °C [2,012 °F] are shown in Figure 2-4(a). The elemental line scans of oxygen, chromium, iron, nickel, molybdenum, and tungsten are presented in Figure 2-4(b). The chromium and oxygen line scan show that the thickness of the external $Cr_2O_3$ scale increased to approximately 14 µm [551.2 µin]. As the measuring probe hit a precipitate in the alloy, an increase in oxygen signal was detected. This indicates that the precipitates observed in Figure 2-4(a) are internally oxidized particles. The maximum depth of oxygen penetration increased to 24 µm [944.9 µin] for this higher temperature.





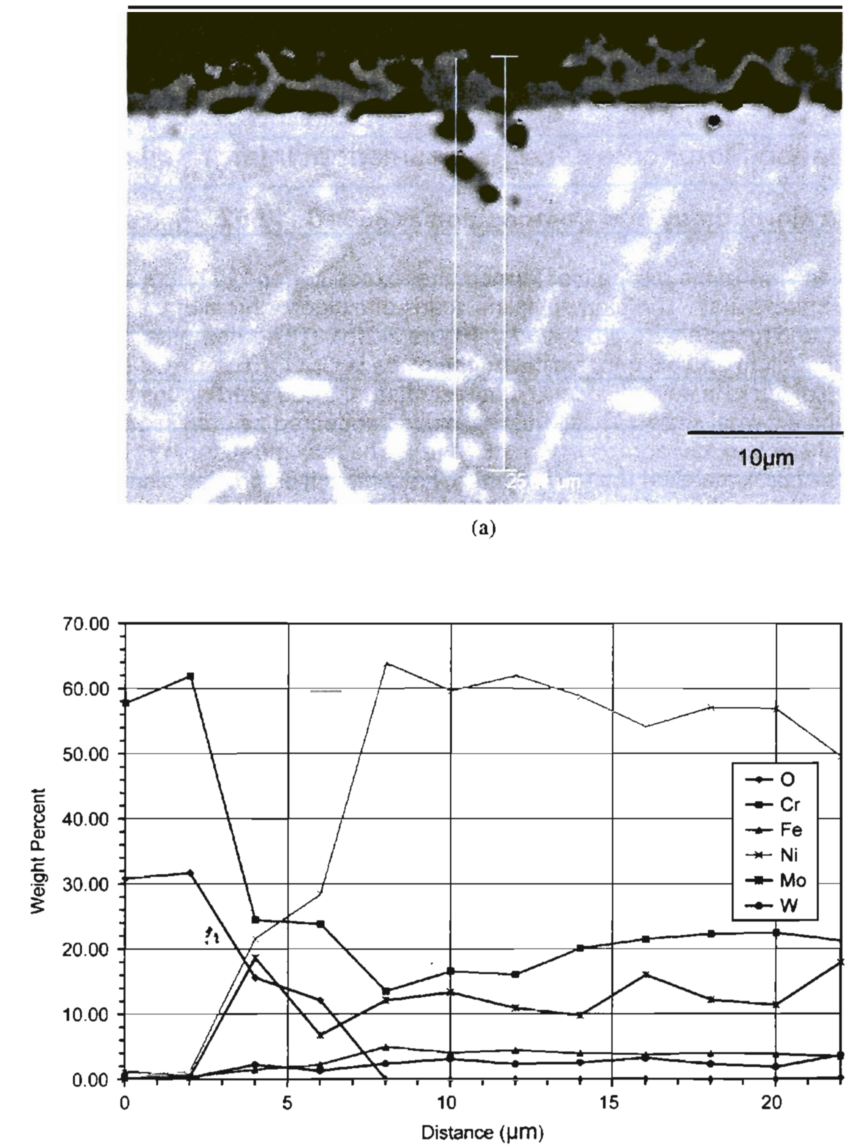


**Figure 2-2. Cross-Section Through the Scale Produced on Alloy 22 After 120 Hours Oxidation at 850 °C [1,562 °F]: (a) Scanning Electron Microscope Backscattered Electron Image; and (b) Concentration Profiles of Oxygen, Chromium, Iron, Nickel, Molybdenum, and Tungsten Along the Line of Traverse Indicated in Figure 2-2(a).**

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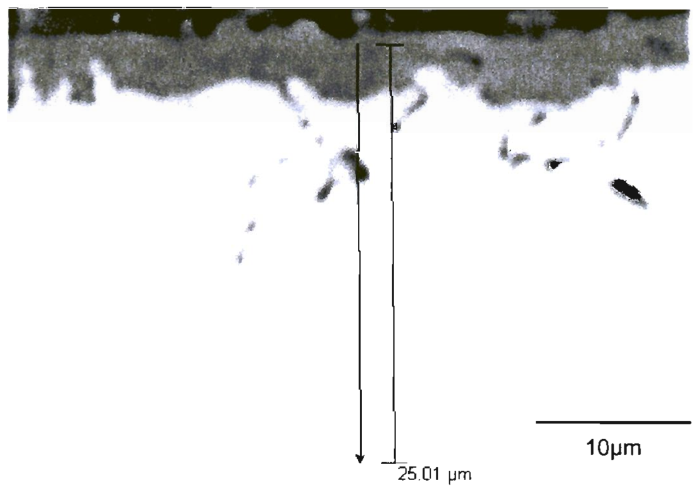
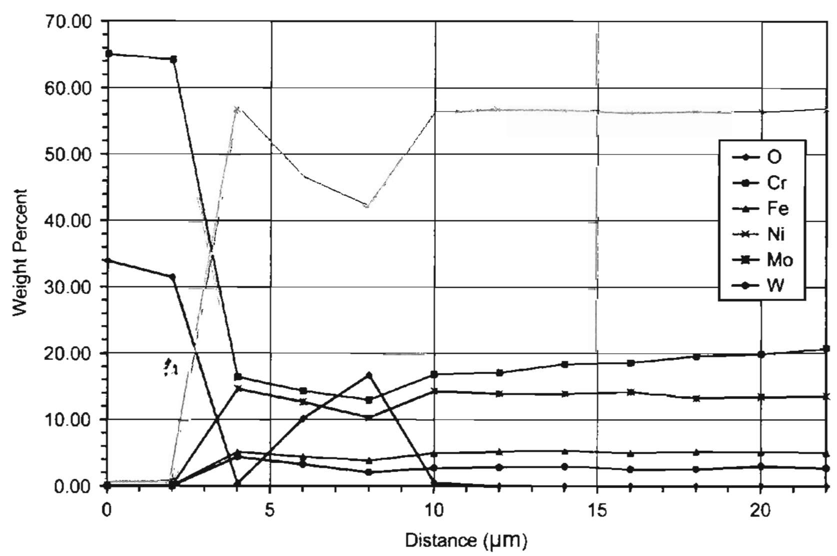



(a)




(b)

**Figure 2-3. Cross-Section Through the Scale Produced on Alloy 22 After 24 Hours Oxidation at 1,100 °C [2,012 °F]: (a) Scanning Electron Microscope Backscattered Electron Image; and (b) Concentration Profiles of Oxygen, Chromium, Iron, Nickel, Molybdenum, and Tungsten Along the Line of Traverse Indicated in Figure 2-3(a)**

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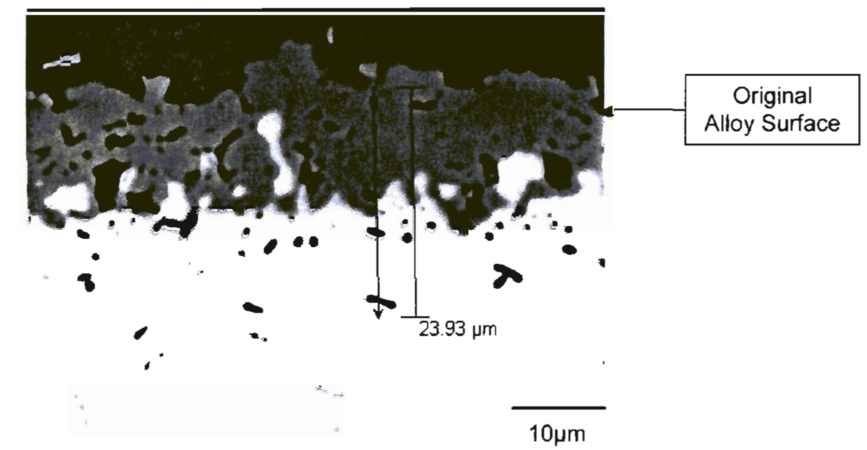
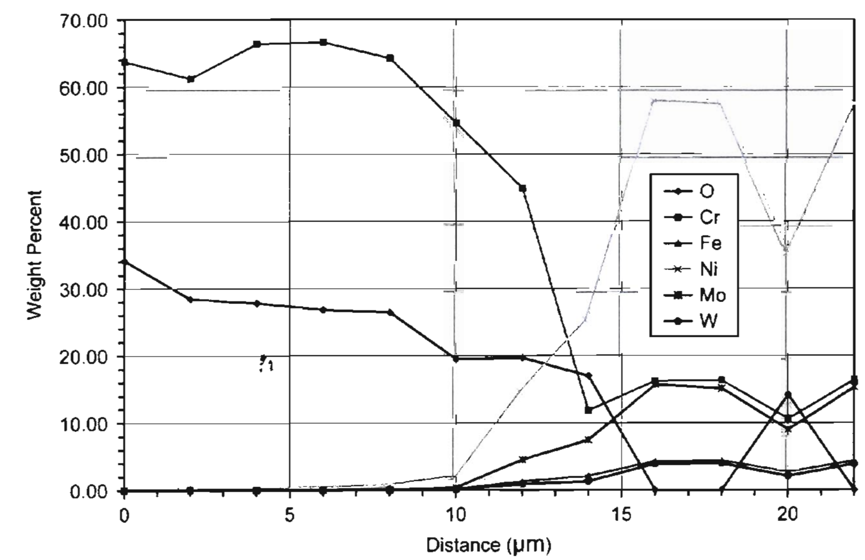



(a)




(b)

**Figure 2-4. Oxide Morphology for Alloy 22 Oxidized in Air for 120 Hours at 1,100 °C [2,012 °F]: (a) Scanning Electron Microscope Backscattered Electron Image; and (b) Concentration Profiles of Oxygen, Chromium, Iron, Nickel, Molybdenum, and Tungsten Along the Line of Traverse Indicated in Figure 2-4(a)**

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To complete the description of oxide morphology, oxygen, chromium, nickel, molybdenum, iron and tungsten x-ray maps are presented in Figure 2-5. The region examined is the same area shown in Figure 2-4(a). The external scale is a continuous layer of $Cr_2O_3$ in contact with the gas environment. The inner scale region is not uniform and is composed of $Cr_2O_3$ scale with isolated islands of unoxidized alloy. The unoxidized alloy contains the metallic elements chromium, iron, nickel, molybdenum, and tungsten, but no oxygen. Numerous internal oxide precipitates are clearly seen underneath the external $Cr_2O_3$ scale.

## 2.5 Analysis of Results and Discussion

Dry-air oxidation of an Alloy 22 engineering barrier is expected to occur when the relative humidity of the repository is less than a critical relative humidity for the initiation of humid-air corrosion. If no drift degradation occurs prior to the thermal pulse, dry-air oxidation is expected to occur beyond 1,000 years. The maximum waste package temperature is expected to be approximately 236 °C [457 °F] if drift degradation occurs (Fedors, et al., 2004; Manepally, et al., 2004). The rate of dry-air oxidation can be modeled by assuming that mass transport of reacting species is limited by solid-state diffusion through a tightly adherent passive $Cr_2O_3$ oxide film.

The oxidation kinetics at 850 °C [1,562 °F] follow a parabolic rate law. The oxidation mechanism is likely predominantly controlled by the outward diffusion of chromium to form the external $Cr_2O_3$ film. The estimated oxidation rate is consistent with the dry-air oxidation model (Bechtel SAIC Company, LLC, 2003a).

Another mode of oxidation degradation of alloys in dry-air environment at elevated temperatures is internal oxidation. Oxygen may diffuse inward and form internal oxides in the alloy matrix or form internal precipitates along grain boundaries. Formation of internal oxides along grain boundaries, also known as intergranular oxidation, has been reported for Fe-21Cr-32Ni alloy after oxidation at 900 °C [1,652 °F] for 3,000 hours (Ahn, 1996; Shida, et al., 1992a,b).

Internal oxidation and intergranular oxidation is more evident at 1,100 °C [2,012 °F] than at 850 °C [1,562 °F]. During early oxidation stages, chromium diffuses outward to form external $Cr_2O_3$ scale at the oxide/gas interface. Oxygen also diffuses inward to form oxide precipitates in the alloy matrix and grain boundaries resulted in metallurgical changes in the region underneath the external scale. Subsequent oxidation involves further oxidation around oxide precipitates in the chromium depletion zone. At 1,100 °C [2,012 °F], thinning of $Cr_2O_3$ scale by vapor loss is significant at the oxide/gas interface (Birks and Meier, 1983; Gulbransen and Jansson, 1970). The thinning of $Cr_2O_3$ scale by vapor loss may contribute to the decreases in parabolic rate constant measurements in oxidation kinetics studies.

The oxidation test results in this study are consistent with the DOE model (Bechtel SAIC Company, LLC, 2003a) that indicate Alloy 22 forms an external $Cr_2O_3$ scale during dry-air oxidation with a diffusion-controlled kinetics. However, the DOE model does not include internal oxidation. Internal oxidation was observed in the current studies at both temperatures of 1,100 °C [2,012 °F] and 850 °C [1,562 °F]. The amounts of internal oxide precipitates decreased markedly as the temperature decreased from 1,100 °C [2,012 °F] to 850 °C [1,562 °F] (see Figures 2-2a and 2-4a). There is not yet sufficient information to extrapolate the internal oxidation results to lower temperatures over extended times. Tests are planned to collect oxide growth rate and internal oxidation data over a range of temperatures. An





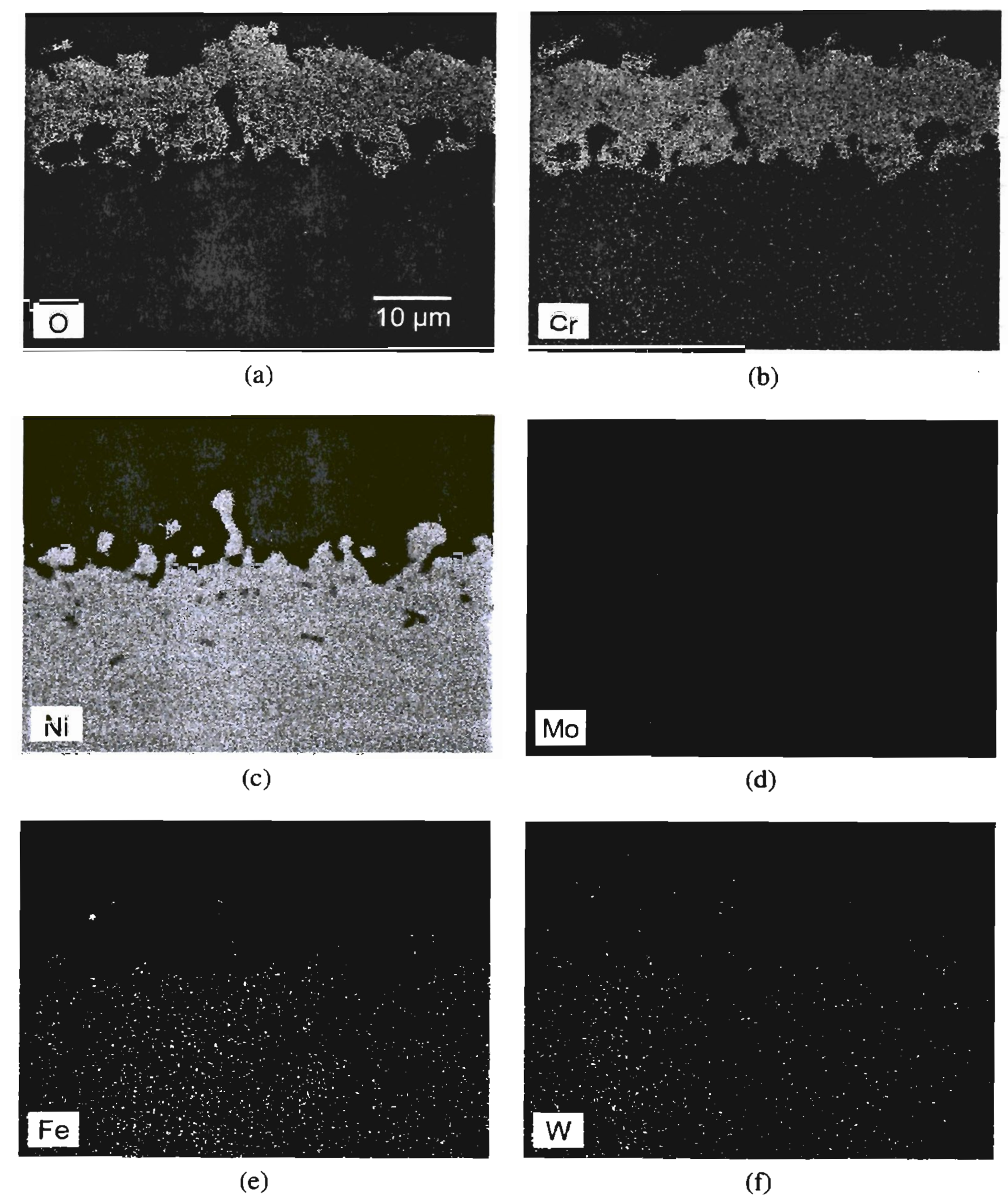


**Figure 2-5. Distribution of Oxygen, Chromium, Iron, Molybdenum, and Tungsten in the Area Outlined in Figure 2-4a: (a) Oxygen X-Ray Map; (b) Chromium X-Ray Map; (c) Nickel X-Ray Map; (d) Molybdenum X-Ray Map; (e) Iron X-Ray Map; and (f) Tungsten X-Ray Map**

assessment of significance of dry-air oxidation at repository temperatures will be made after data at lower temperatures have been acquired and analyzed. For example, oxide growth rates could be estimated at higher temperatures and a trend could be established as a function of temperature. Also, the oxygen penetration depth could be estimated as a function of time and temperature, delineating functions that could be extrapolated to lower temperatures that are relevant to repository conditions. These temperature-time trends could be employed to estimate the extent of alloy change in the neighborhood of the metal-oxide interface by the time an aqueous environment is established and aqueous corrosion initiated. It is argued elsewhere (Pensado, et al., 2002) and in Chapter 5 that passive dissolution, in the presence of benign environmental conditions, can only be disturbed by processes inducing compositional alloy changes in the region close to the metal-oxide interface. If this alloy does not change (i.e., its composition remains close to the initial bulk alloy composition), passive oxides are expected to form, yielding corrosion rates comparable to those determined under the controlled conditions discussed in this report. Thus, if dry-oxidation does not induce significant compositional changes to the alloy close to the metal-oxide interface, passive dissolution is expected to prevail after the establishment of benign aqueous environments in contact with waste package materials. Estimating the extent of anticipated changes to the alloy composition in the neighborhood of the metal-oxide interface is important to enhance confidence that passive dissolution can be sustained for prolonged periods, in the absence of environmental conditions leading to localized corrosion.



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**Initial Scientific Notebook Entry for Dry-Air Oxidation, Humid Air Oxidation, Passive Film.**

**Title:** Isothermal oxidation tests, cyclic oxidation tests, dry-air oxidation with deposited salt, oxygen 16/oxygen 18 experiments, microstructure analysis.

**Test Performed by:** Kuang-Tsan Kenneth Chiang

**Objective:** Study the oxidation kinetics, oxide morphology, and mechanism of passive film formation in dry air, humid air and thermal cycling conditions. Study the effect of fabrication processes such as welding and post-weld heat treatment on passive film formation.

**Equipment:** High Temperature Microbalance TherMax 700, Lindberg Furnace, Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Analysis (EDX), X-ray Diffraction (XRD), Secondary Ion Mass Spectroscopy (SIMS) of oxygen 16/oxygen 18 oxidized specimens will be performed by a qualified supplier through a purchase order.

**Materials:** Alloy C-22, Alloy 825

**Specimen Specifications:** ASTM G54-77 for determining total depth of attack for static oxidation testing.

**Measurement Parameters:** Temperature of oxidation treatment, weight change after oxidation and thermal cycling.

**Required Level of Accuracy:** Temperature ±3°C for oxidation studies, weight measurement ±.005 mg.

**Uncertainty and Source of Error:** Duplicated oxidation specimens will be tested for each oxidation conditions.

Also See pg# 1



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