

Project No. 06002.01.342
14002.01.352

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Initial Entry - SN 840

Project Identification: 20.06002.01.342

Scientific notebook 840 is a continuation of SN 790, which is concerned with the interaction between magma and waste package materials¹.

The main objective relevant to this notebook is the evaluation of the corrosion and mechanical degradation of alloy C-22 when subjected to high temperatures and molten rock material. The approach for monitoring these effects involves exposing the alloy in differing machined forms to magma for specific lengths of time within a high temperature oven. The samples are then analyzed with varying methods of microscopy in order to understand the extent of oxidation and degradation.

Further information is documented in the following entries of this notebook along with the equipment used, procedures followed and data collected.

Individuals involved:

- Kenneth Chiang (Primary Holder)
- Mark Silver M.S.

¹ Chiang, K.T., et al. CNWRA SN 790. San Antonio: CNWRA, 2006.

Task assigned to: Mark Silver

Objective: Monitor the extent of oxidation and corrosion of alloy C-22 when exposed to high temperatures.

Equipment/Materials used:

- Hastelloy C-22 coupons
Heat # : 2277-3-3266
Major element composition: 58% Ni, 22% Cr, 13% Mo, 4% Fe, 3% W (estimated by mass)
Machined to approximately 1.25cm x 1.44cm x 0.15cm
With a 0.16cm diameter hole approximately 0.33cm below the center of the width of the coupon
- Alumina crucibles, 1" diameter (2)
- 50mL glass beakers (4)
- Acetone in a squirt bottle
- Platinum wire
- Steel tongs
- Tweezers
- Heat resistant safety gloves
- Fisher brand twist-tie sample bags, small size
- Cold epoxy mounting kit
- 600 grit adhesive backed 8" diameter abrasive discs
- Buehler Ecomet 3 variable speed grinder/polisher
120 volt
490 Max RPM
Cat # : 60-1960-160
sn: 462-A3P-0208
- Buehler Isomet Plus precision wet cut-off saw
115 volt
4500 Max RPM
Cat # : 11280-160
sn: 452-I5P-835
- Oster dryer
1200 Watt
Service # : 269-01E
- Fisher Scientific FS 14 ultrasonic cleaner
- CM Inc. Rapid Temp furnace
Max temp. 1700°C
sn: 98002-24
Calibration due: Not required
- Lindberg oven
Max temp. 1200°C
Model # : 51333
sn: 909172
Calibration due: Not required
- Omega microprocessor thermometer
Model # : HH22
sn: T94140
Calibration due: 05/13/07
- Thermocouple
sn: 335
Calibration due: 05/25/07
- Starrett electronic digital caliper
Model # : 721
sn: 03031512
Calibration due: 03/02/07
- Sartorius Genius scale
Mass range: 0.00001g to 200g
sn: 12809099
Calibration due: 05/08/07

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Measurement parameters/Precision requirements:

The masses of alloy C-22 samples are to be measured to the nearest milligram, and the dimensions of such samples measured to the nearest thousandth of a centimeter. Temperature measurements will be recorded to the nearest degree centigrade.

Identified sources of error:

- Physical differences in duplicate sample specimens
- Chemical differences in duplicate sample specimens
- Samples which are unclean
- Mass loss due to spalling
- Preferential exposure of oxidizing surfaces

Procedure followed:

1. Preparation of Alloy C-22 coupons:

The variable speed grinder/polisher is used to polish the coupons to 600 grit on all surfaces so that each coupon is identical and all machine scratches are removed. The dimensions of each specimen are then measured to the thousandth of a centimeter with the digital caliper and recorded along with the sample identification. Each sample is submerged in acetone within a separate 50mL beaker and placed in the ultrasonic cleaner for five minutes. The sample coupons are then removed with tweezers, as they should not be touched by hand past the cleaning step, and placed under the dryer until dry. Care should be taken to ensure that the identity of each coupon is not lost. Mass measurements of the clean and dry coupons are then taken to the nearest milligram and the samples are placed into labeled sample bags.

2. Heating the alloy:

The Rapid Temp furnace is set and allowed to achieve the desired temperature before introducing the samples. When the temperature becomes stable, it is checked using a microprocessor thermometer with a thermocouple attachment. The furnace setpoint and true temperature are recorded. Platinum wire is threaded through the holes in the coupons and used to balance them within the alumina crucibles so that there is no direct contact between the samples and the sides of the crucibles. Heat resistant gloves are worn while using tongs to gently place the crucibles within the furnace and the precise time is noted. After a specified duration, the samples are removed and immediately placed into the Lindberg oven already heated to 600°C to incrementally cool for at least one hour (to prevent thermal shock to the samples and crucibles) and the time of this sample transfer is recorded. The samples are then removed and allowed to cool to room temperature.

3. Oxidation and corrosion analysis:

A visual description of the heat treated alloy coupons is recorded along with the final mass of the coupons and the total mass of all material spalled off the coupon. The oxidized samples may then be analyzed and photographed at 1000x and 5000x magnifications with the use of a scanning electron microscope (located in division 18 facilities, Mechanical & Materials Engineering). The samples are then mounted using a cold epoxy mounting kit and cut preferentially with the wet cut-off saw to reveal specific cross sections of the oxidized alloy so that further examination can ensue.

Material Analyzed:

C-22

Sample Dimensions and Mass:

Sample Identity: OX1
 Width: 1.240 cm
 Length: 1.437 cm
 Thickness: 0.157 cm
 Surface Area: 4.443 cm² (calculated)
 Initial Mass: 2359 mg

Sample Identity: OX2
 Width: 1.268 cm
 Length: 1.437 cm
 Thickness: 0.151 cm
 Surface Area: 4.497 cm² (calculated)
 Initial Mass: 2326 mg

Sample Identity: OX3
 Width: 1.276 cm
 Length: 1.441 cm
 Thickness: 0.146 cm
 Surface Area: 4.504 cm² (calculated)
 Initial Mass: 2262 mg

Sample Identity: OX4
 Width: 1.268 cm
 Length: 1.441 cm
 Thickness: 0.153 cm
 Surface Area: 4.520 cm² (calculated)
 Initial Mass: 2398 mg

Sample Identity: OX5
 Width: 1.240 cm
 Length: 1.384 cm
 Thickness: 0.154 cm
 Surface Area: 4.278 cm² (calculated)
 Initial Mass: 2242 mg

Sample Identity: OX6
 Width: 1.241 cm
 Length: 1.435 cm
 Thickness: 0.144 cm
 Surface Area: 4.365 cm² (calculated)
 Initial Mass: 2158 mg

Material Analyzed:

C-22 6 January 2007 M.S.

Sample Dimensions and Mass:

Sample Identity: OX7
 Width: 1.256 cm
 Length: 1.422 cm
 Thickness: 0.149 cm
 Surface Area: 4.405 cm² (calculated)
 Initial Mass: 2257 mg

Sample Identity: OX8
 Width: 1.257 cm
 Length: 1.437 cm
 Thickness: 0.156 cm
 Surface Area: 4.491 cm² (calculated)
 Initial Mass: 2372 mg

Sample Identity: OX9
 Width: 1.261 cm
 Length: 1.416 cm
 Thickness: 0.156 cm
 Surface Area: 4.445 cm² (calculated)
 Initial Mass: 2355 mg

Sample Identity: OX10
 Width: 1.261 cm
 Length: 1.420 cm
 Thickness: 0.158 cm
 Surface Area: 4.468 cm² (calculated)
 Initial Mass: 2391 mg

Sample Identity: OX11
 Width: 1.254 cm
 Length: 1.433 cm
 Thickness: 0.153 cm
 Surface Area: 4.453 cm² (calculated)
 Initial Mass: 2328 mg

Sample Identity: OX12
 Width: 1.243 cm
 Length: 1.427 cm
 Thickness: 0.156 cm
 Surface Area: 4.419 cm² (calculated)
 Initial Mass: 2346 mg

Material Analyzed:

C-22

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Sample Dimensions and Mass:

Sample Identity: OX13
 Width: 1.250 cm
 Length: 1.425 cm
 Thickness: 0.150 cm
 Surface Area: 4.400 cm² (calculated)
 Initial Mass: 2263 mg

Sample Identity: OX14
 Width: 1.248 cm
 Length: 1.447 cm
 Thickness: 0.151 cm
 Surface Area: 4.461 cm² (calculated)
 Initial Mass: 2341 mg

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Temperature and Duration Specifications:

Sample OX1

Furnace setpoint: 1000 °C
 Furnace temperature: 1029.0 °C
 Duration of exposure: 20 hrs.
 Placed in furnace: 12:47 pm 01/05/07
 Removed from furnace: 8:47 am 01/06/07

Visual description: *dull oxidized surface, but dimensions and integrity maintained. No spall.*

Final mass of coupon: 2329 mg
 Mass of spalled fragments: 0 mg
 Total mass: 2329 mg

Sample OX2

Furnace setpoint: 1000 °C
 Furnace temperature: 1029.0 °C
 Duration of exposure: 48 hrs.
 Placed in furnace: 12:47 pm 01/05/07
 Removed from furnace: 12:47 pm 01/07/07

Visual description: *dull oxidized surface, but dimensions and integrity maintained. No spall.*

Final mass of coupon: 2362 mg
 Mass of spalled fragments: 0 mg
 Total mass: 2362 mg

Sample OX3

Furnace setpoint: 1300 °C
 Furnace temperature: 1301.5 °C
 Duration of exposure: 20 hrs.
 Placed in furnace: 2:04 pm 01/08/07
 Removed from furnace: 10:04 am 01/09/07

Visual description: *alloy completely consumed by oxidation with relatively coarse crystalline oxide crystals throughout. Fragmented oxide.*

Final mass of coupon: 643 mg
 Mass of spalled fragments: 1939 mg
 Total mass: 2582 mg

Sample OX4

Furnace setpoint: 1300 °C
 Furnace temperature: 1301.5 °C
 Duration of exposure: 48 hrs.
 Placed in furnace: 2:04 pm 01/08/07
 Removed from furnace: 2:04 pm 01/10/07

Visual description: *completely consumed by oxidation. Interlocking oxide crystals held material together, though dimensions became larger.*

Final mass of coupon: 2689 mg
 Mass of spalled fragments: 7 mg
 Total mass: 2696 mg

Sample OX5

Furnace setpoint: 1100 °C
 Furnace temperature: 1120.9 °C
 Duration of exposure: 20 hrs.
 Placed in furnace: 4:01 pm 01/10/08
 Removed from furnace: 1:01 pm 01/11/07

Visual description: *a brown oxide coating of various thickness covers the coupon though integrity is maintained. No spall.*

Final mass of coupon: 2247 mg
 Mass of spalled fragments: 0 mg
 Total mass: 2247 mg

Sample OX6

Furnace setpoint: 1100 °C
 Furnace temperature: 1120.9 °C
 Duration of exposure: 48 hrs.
 Placed in furnace: 4:01 pm 01/10/08
 Removed from furnace: 4:01 pm 01/12/07

Visual description: *a brown oxide coating of various thickness covers the coupon though integrity is maintained. No spall.*

Final mass of coupon: 2161 mg
 Mass of spalled fragments: 0 mg
 Total mass: 2161 mg

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Sample OX 7

Furnace setpoint: 1200 °C
 Furnace temperature: 1212.7 °C
 Duration of exposure: 20 hrs.
 Placed in furnace: 6:53 pm 01/15/07
 Removed from furnace: 3:03 pm 01/16/07

Visual description: *a brown oxide coating covers the coupon and is loose at the surface. The edges of the coupon have become irregular and a small amount of spall was collected from the coupon.*

Final mass of coupon: 2232 mg
 Mass of spalled fragments: 38 mg
 Total mass: 2270 mg

Sample OX 8

Furnace setpoint: 1200 °C
 Furnace temperature: 1212.7 °C
 Duration of exposure: 48 hrs.
 Placed in furnace: 6:53 pm 01/15/07
 Removed from furnace: 6:53 pm 01/17/07

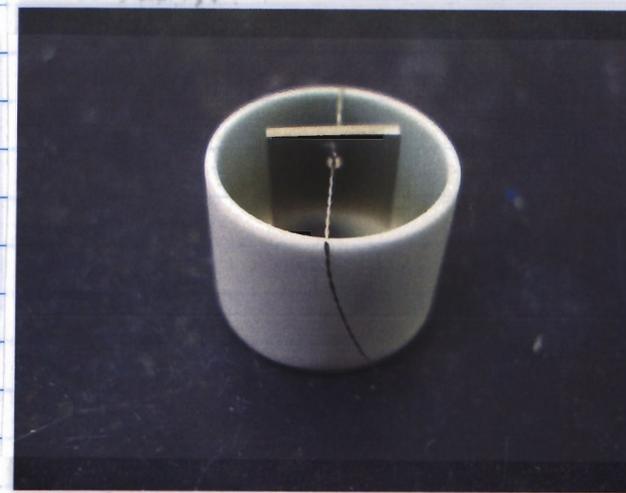
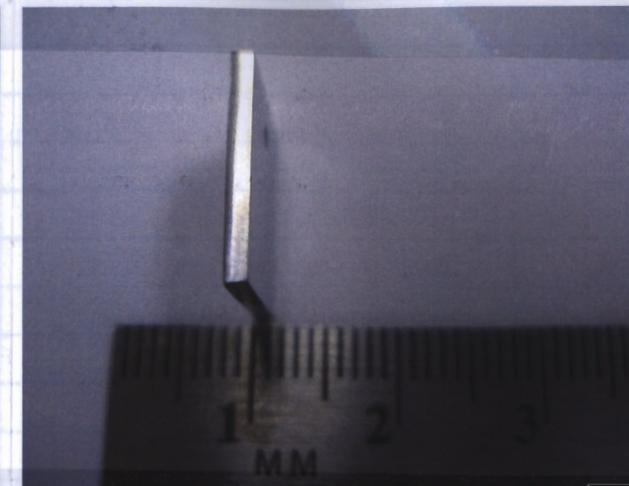
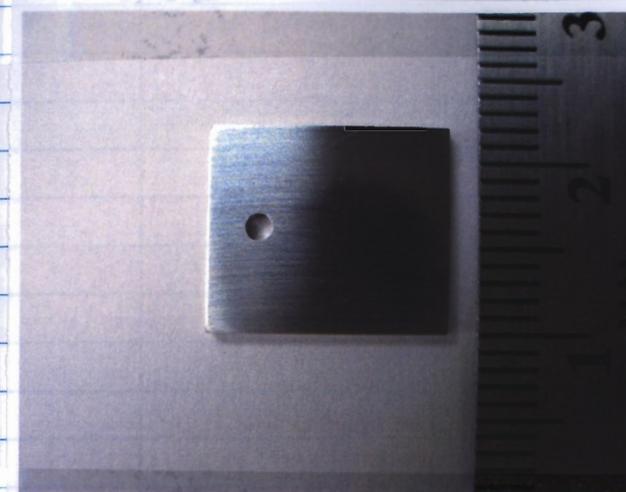
Visual description: *a brown oxide coating covers the coupon. Coupon edges are irregular. a larger amount of spall was collected from the coupon.*

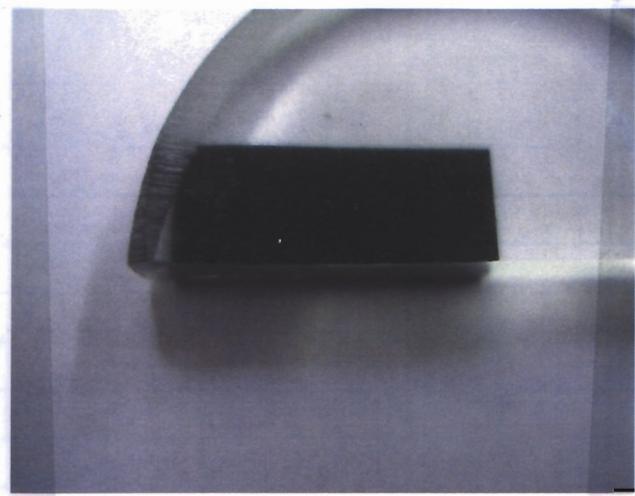
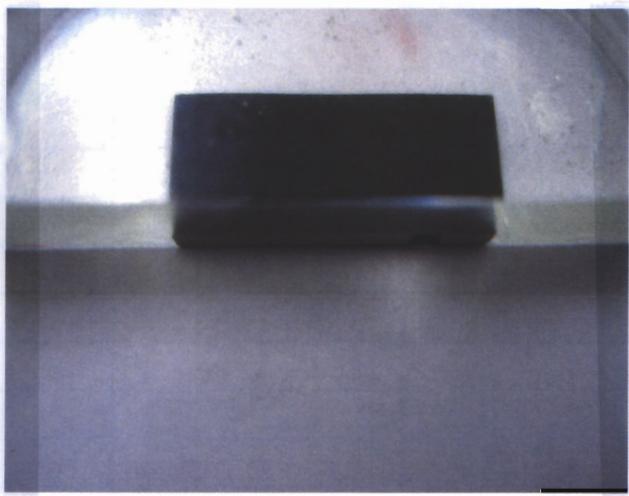
Final mass of coupon: 2294 mg
 Mass of spalled fragments: 105 mg
 Total mass: 2399 mg

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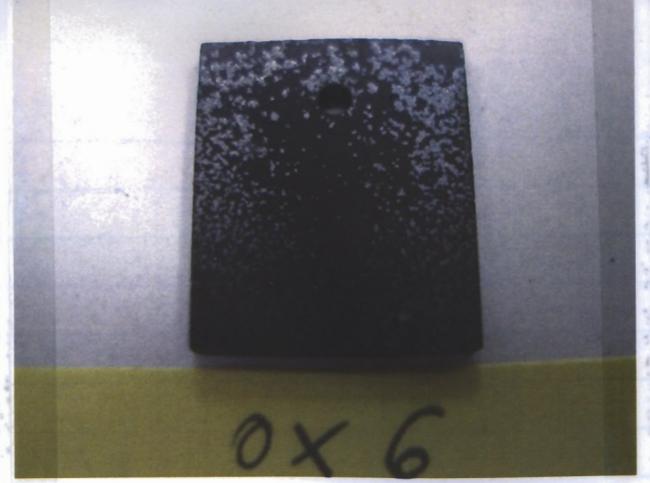
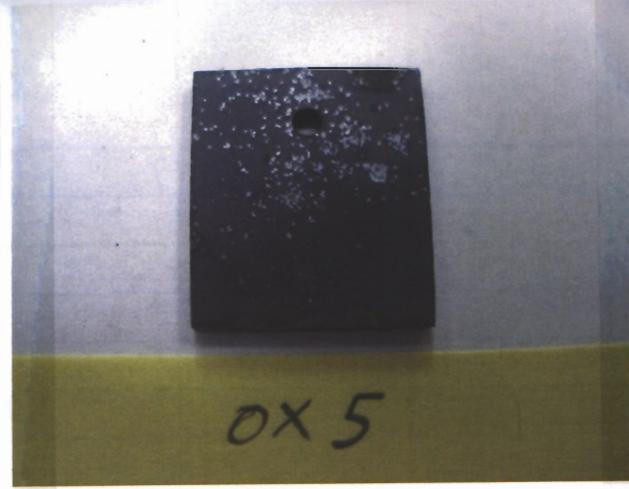
General coupon appearance before and during exposure to high temperatures.





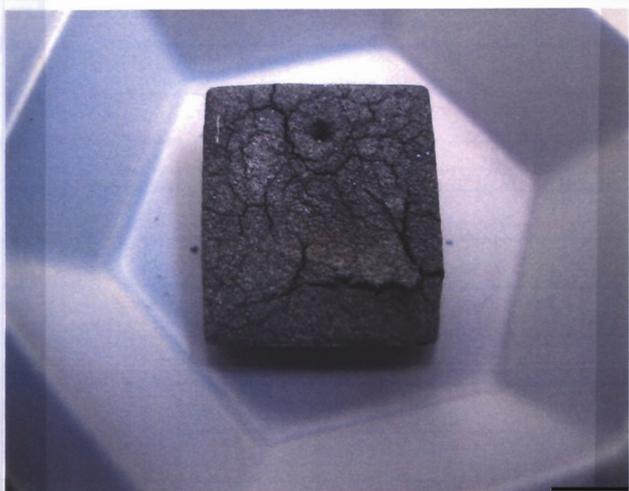
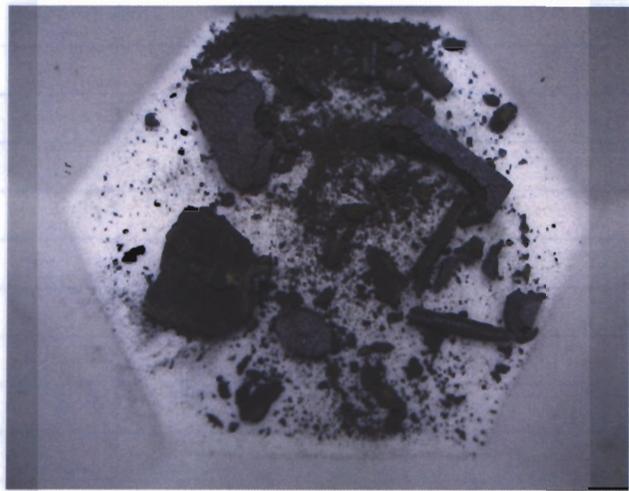
OX1 after 20 hours @ 1029.0°C
(cut and partially mounted)

OX2 after 48 hours @ 1029.0°C
(cut and partially mounted)



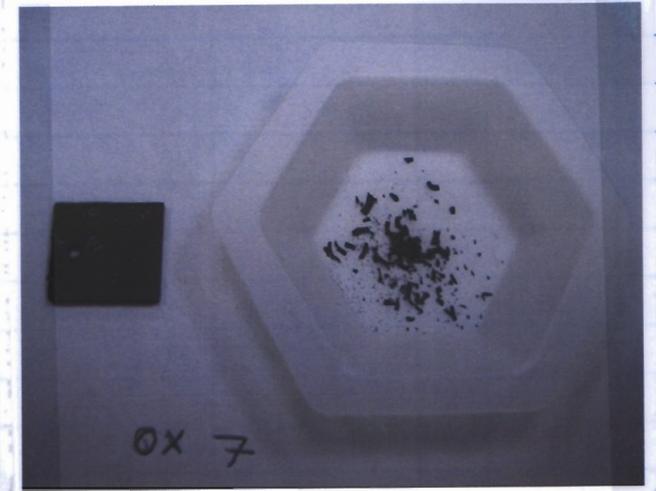
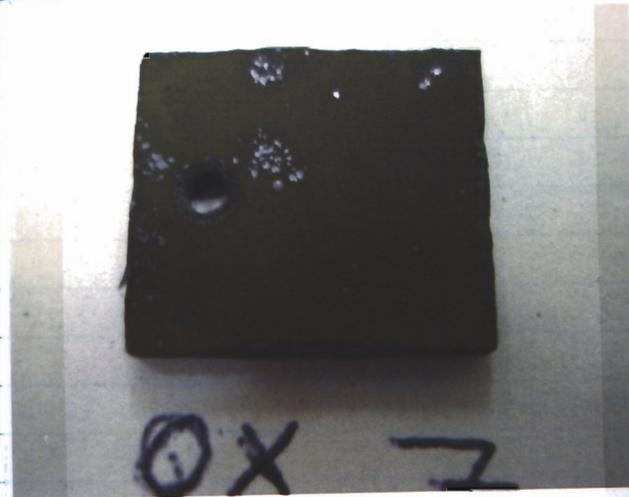
OX5 after 21 hours @ 1120.9°C

OX6 after 48 hours @ 1120.9°C



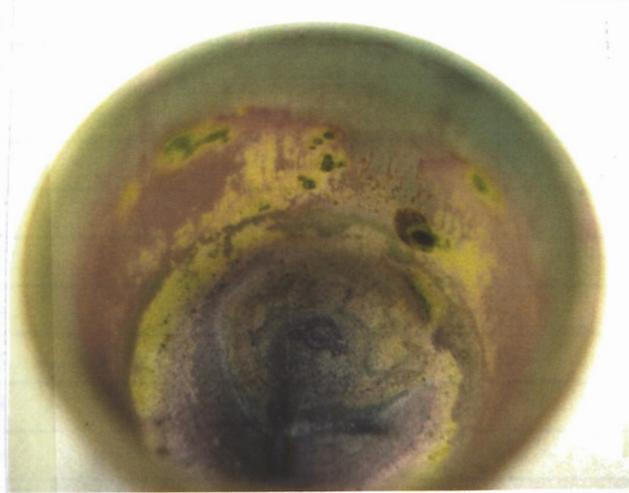
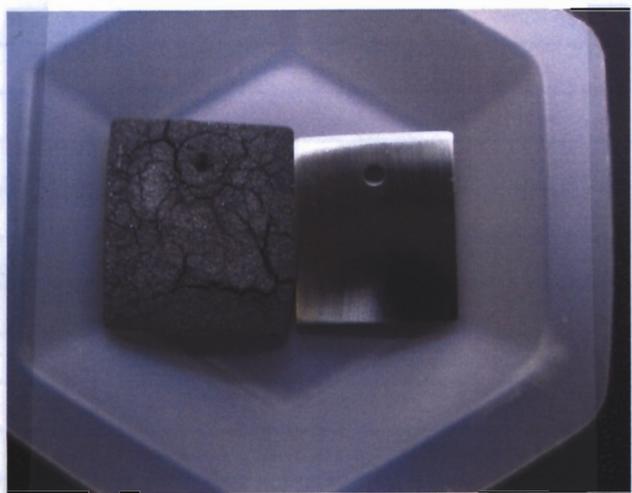
OX3 after 20 hours @ 1301.5°C

OX4 after 48 hours @ 1301.5°C



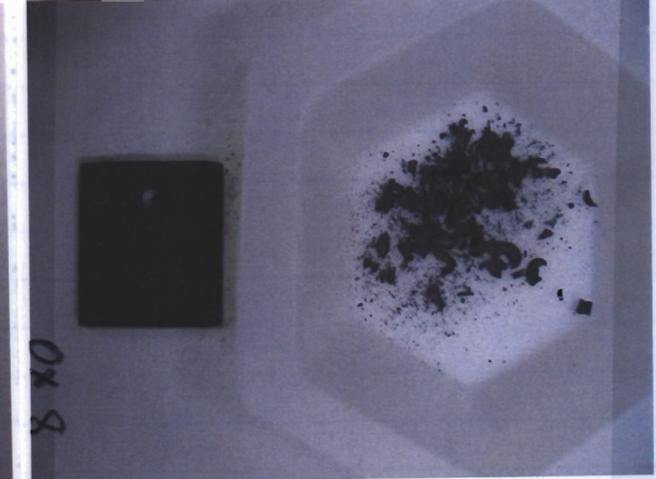
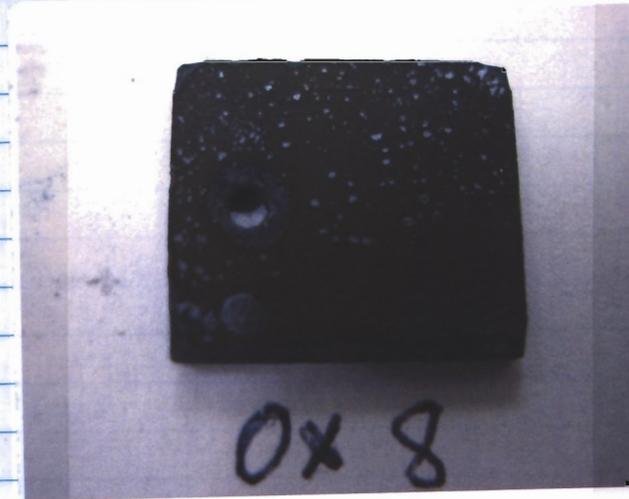
OX7 after 20 hours, 10 minutes @ 1212.7°C

shown with spall



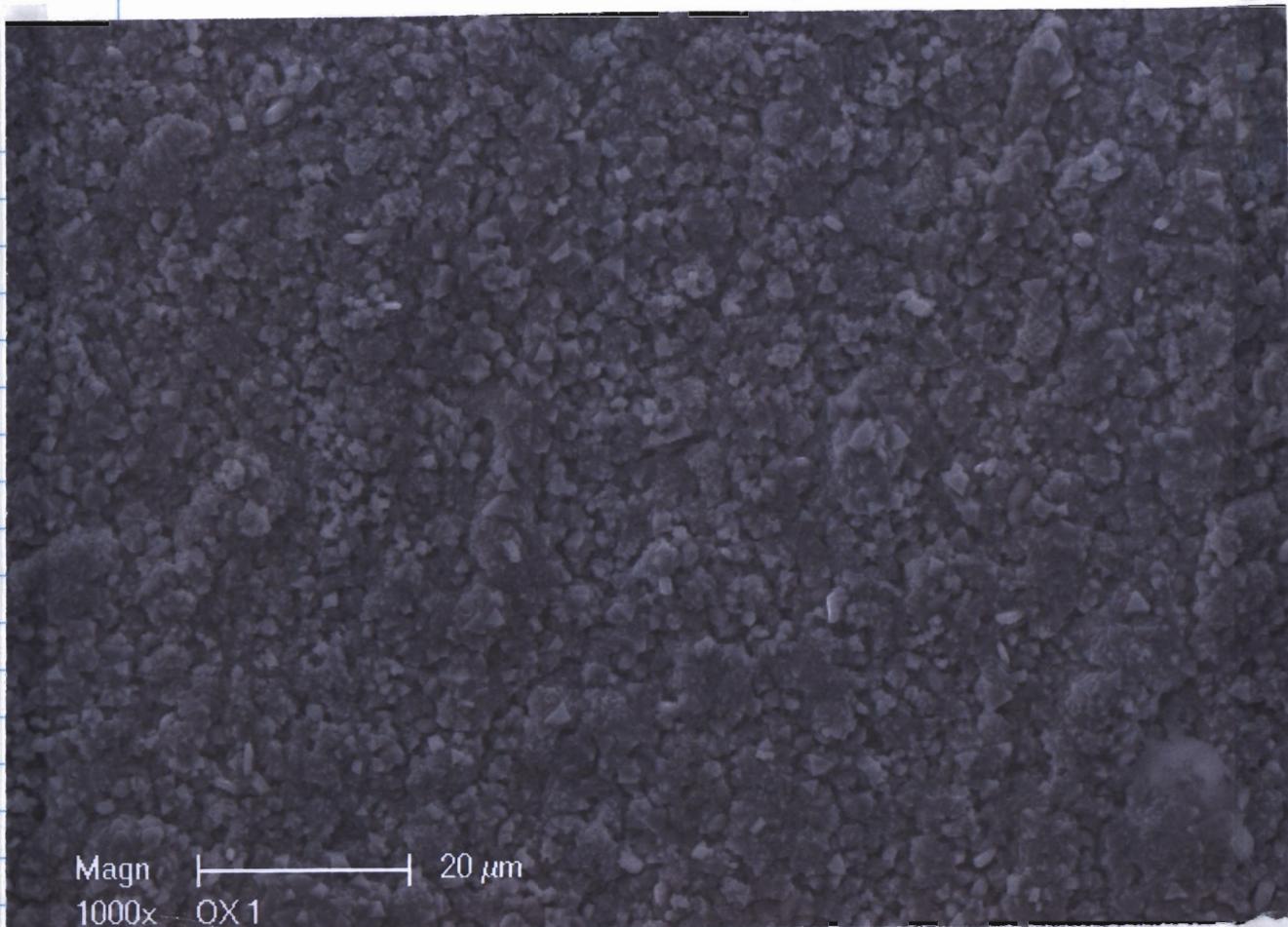
OX4 comparison with un-heated coupon

Colorful Ni and Cr oxides within crucible

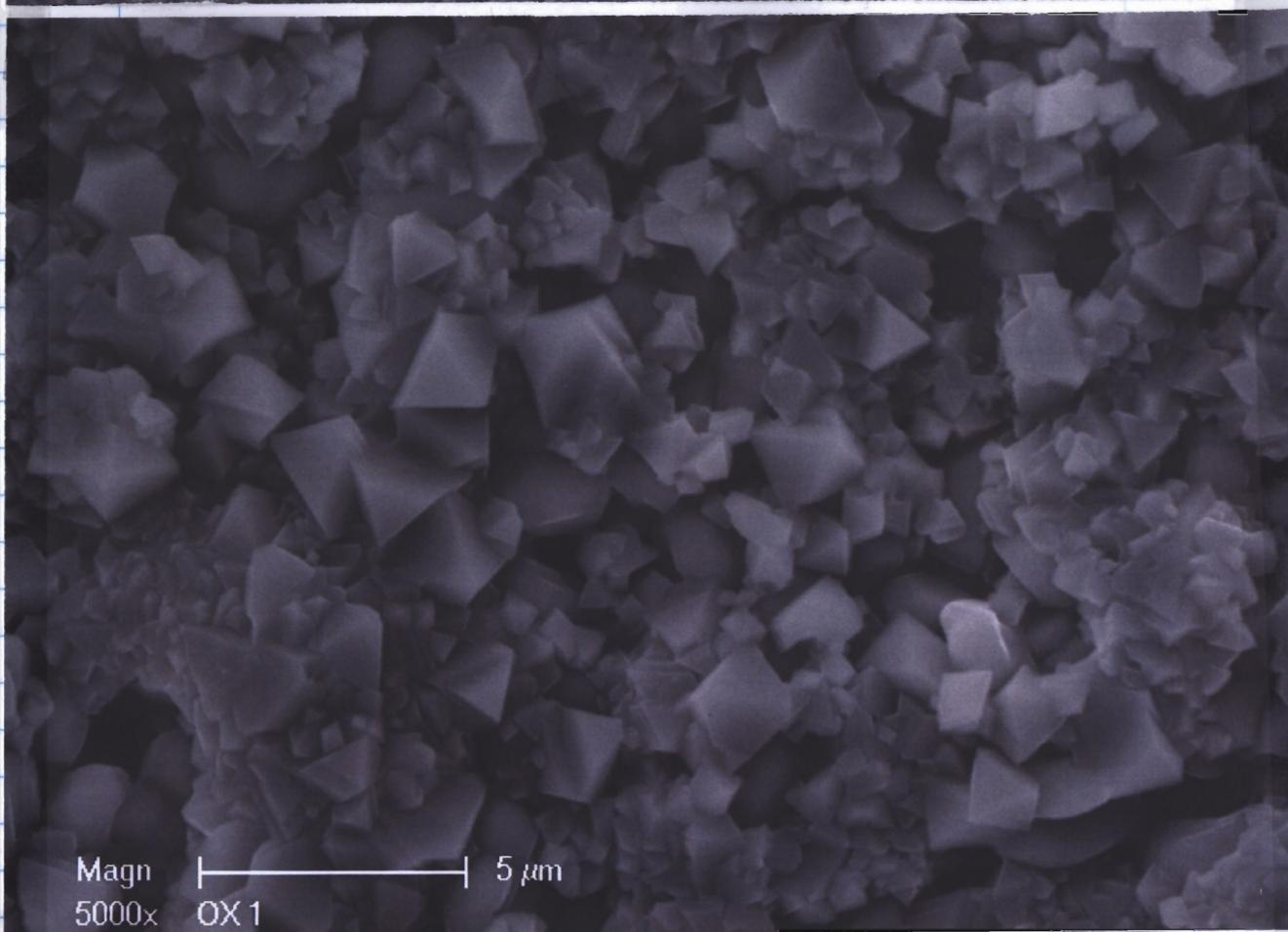


OX8 after 48 hours @ 1212.7°C

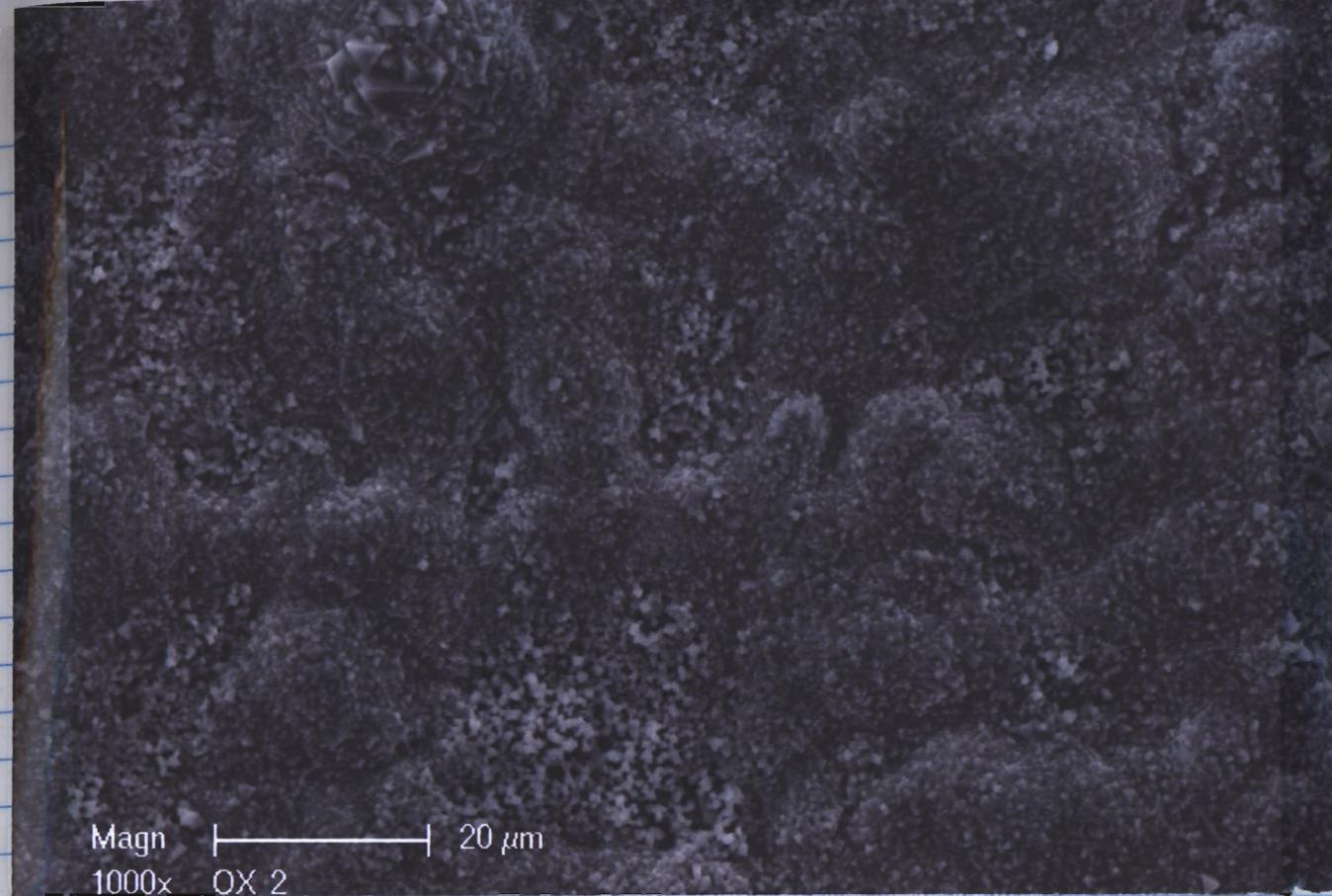
shown with spall



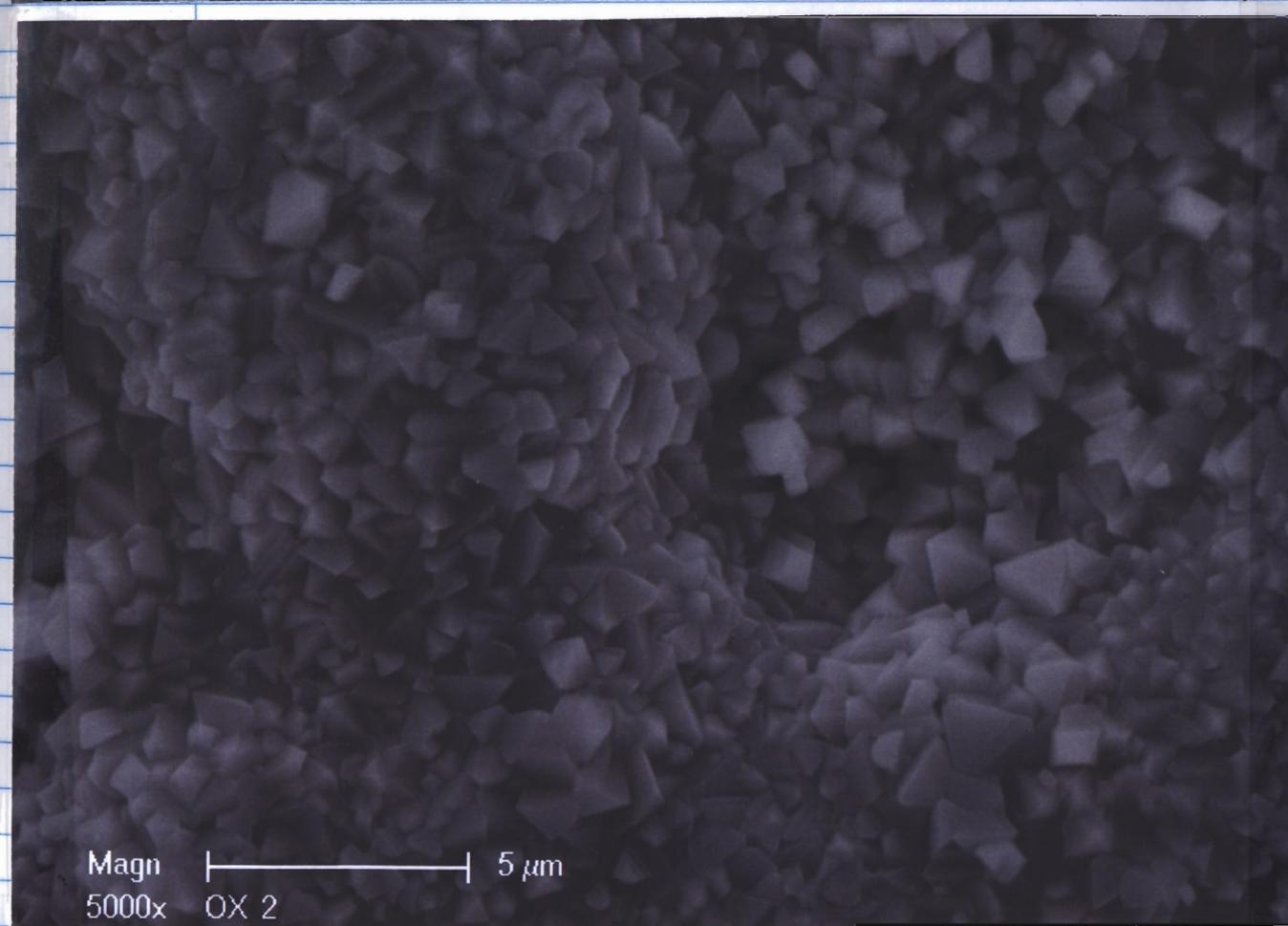
Magn |-----| 20 μ m
1000x OX 1



Magn |-----| 5 μ m
5000x OX 1

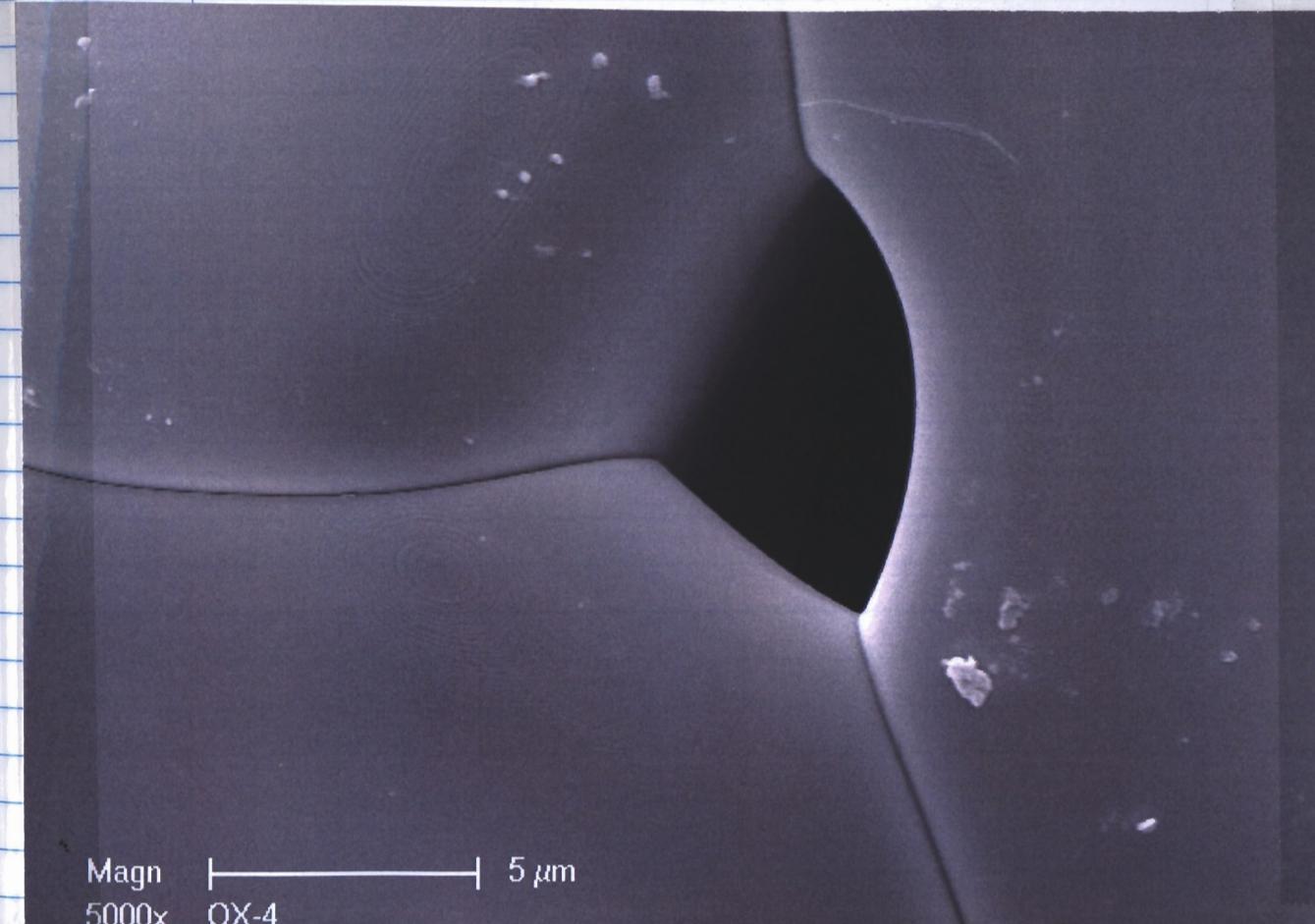
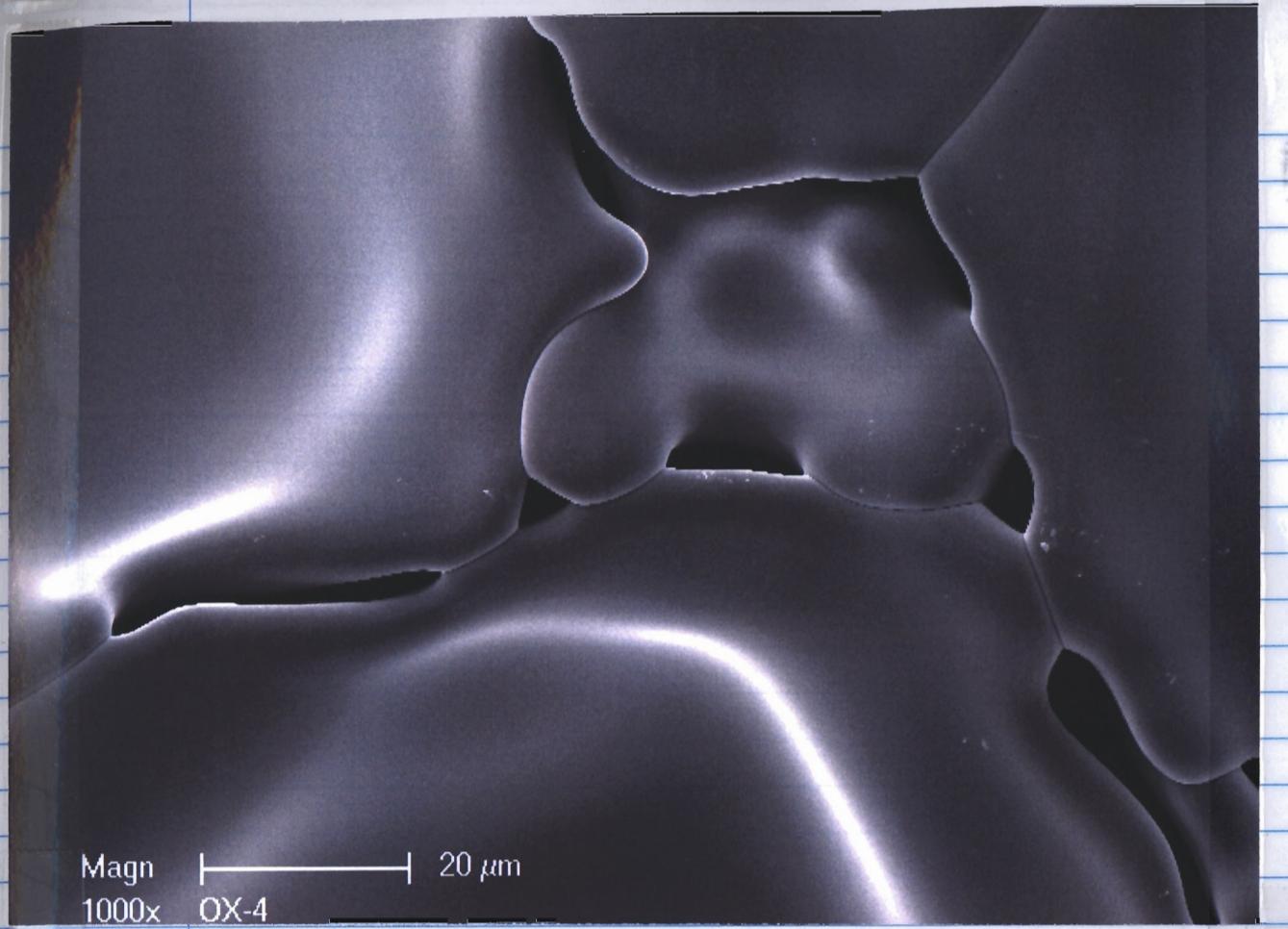
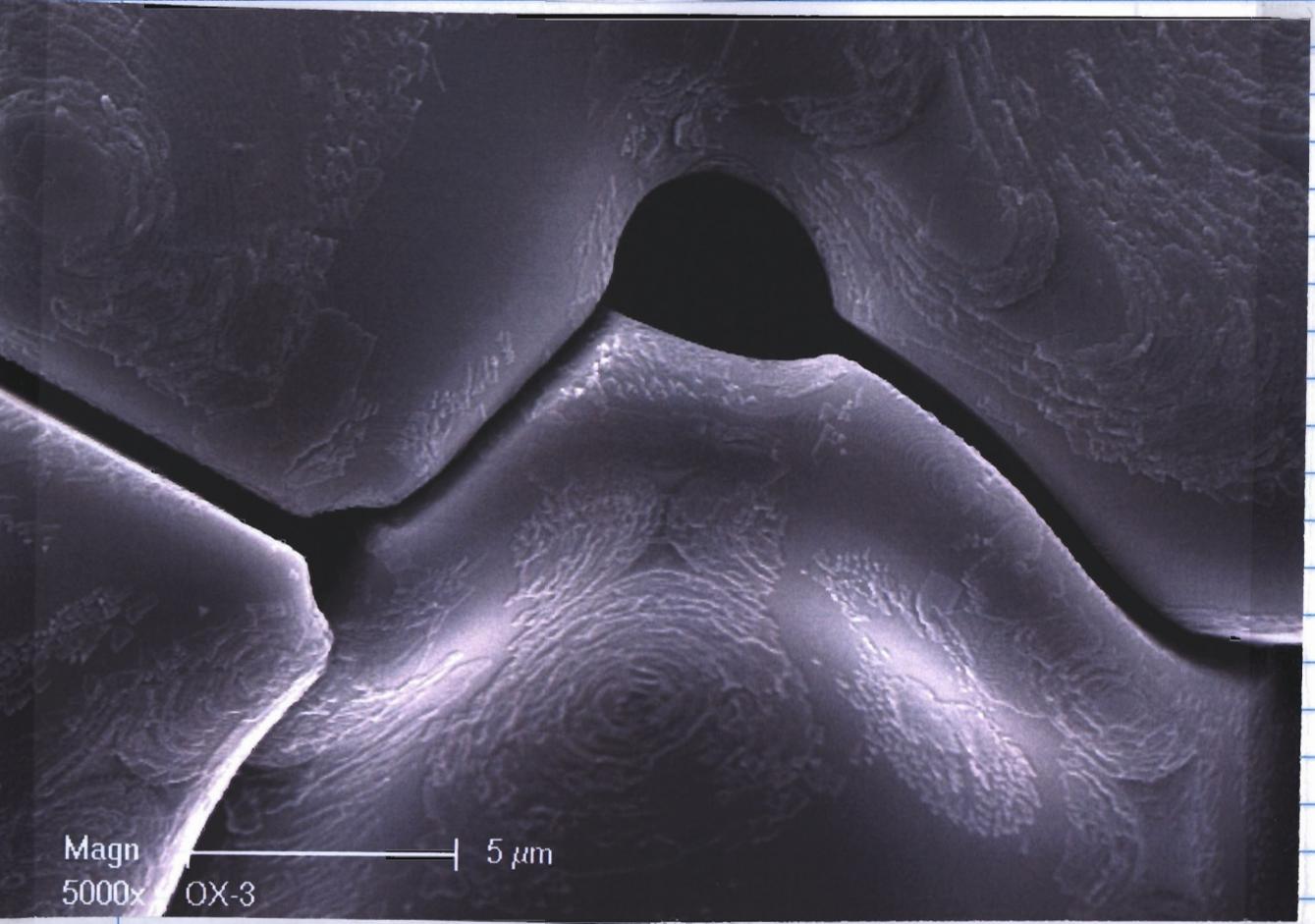
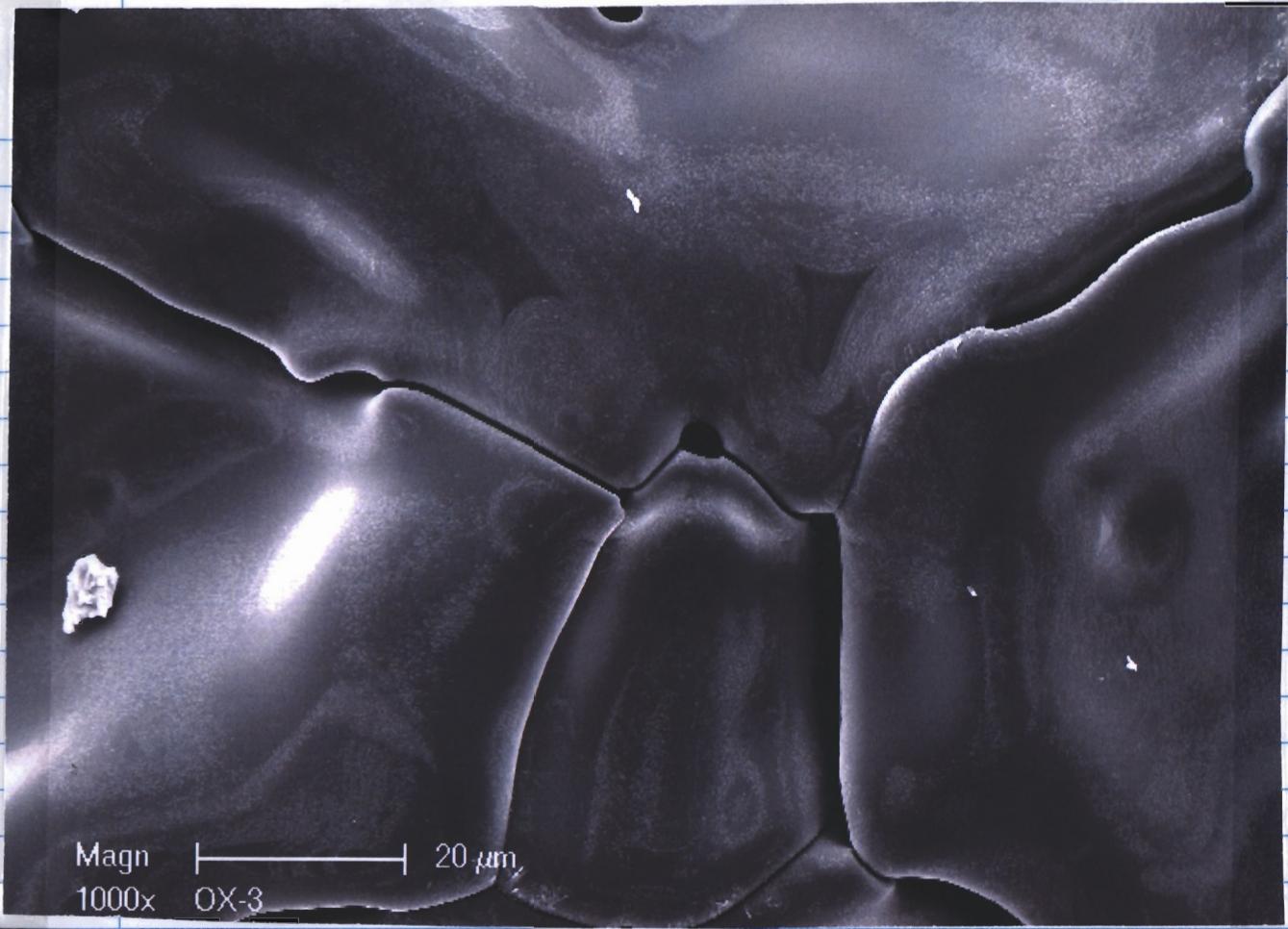


Magn |-----| 20 μ m
1000x OX 2

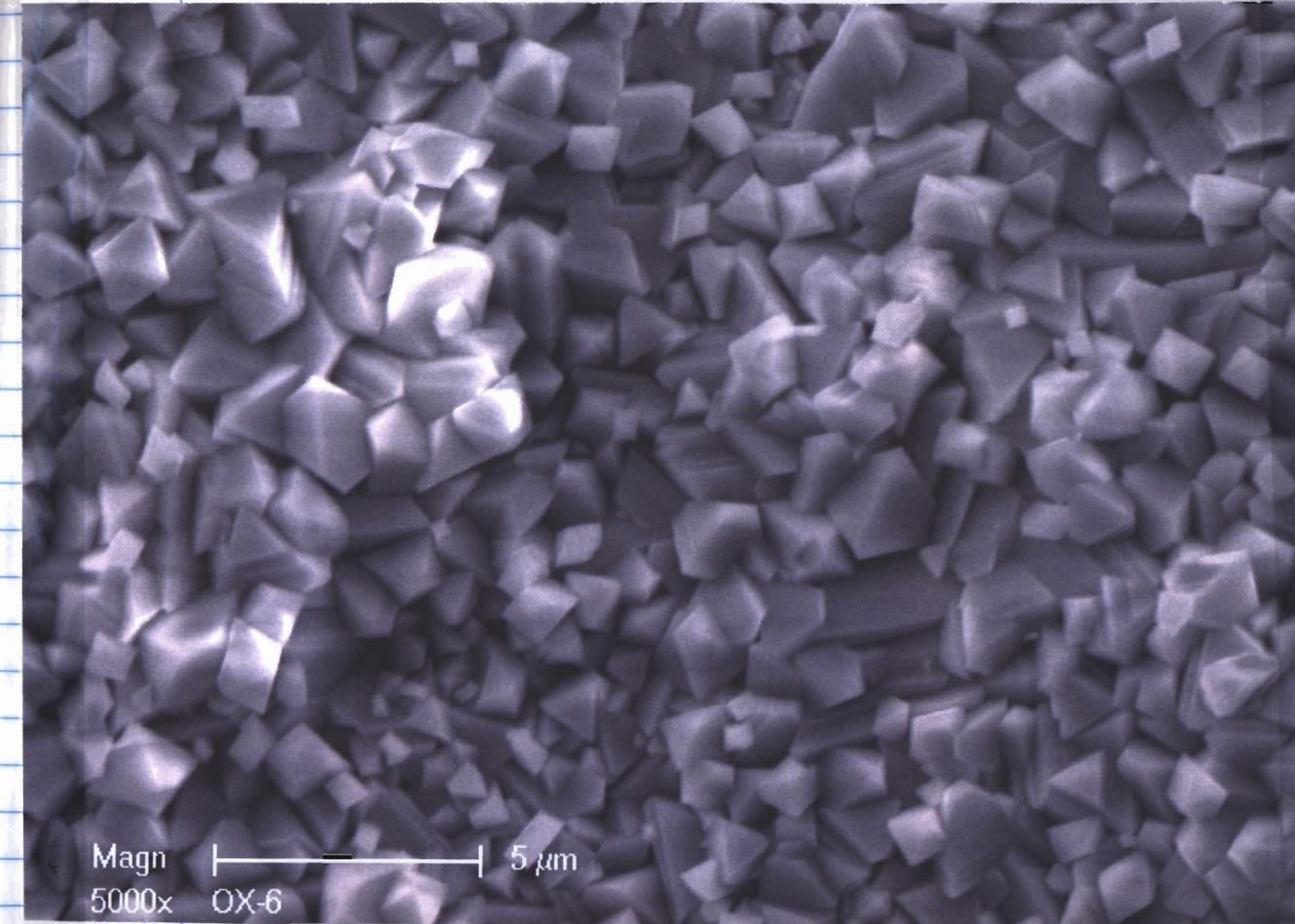
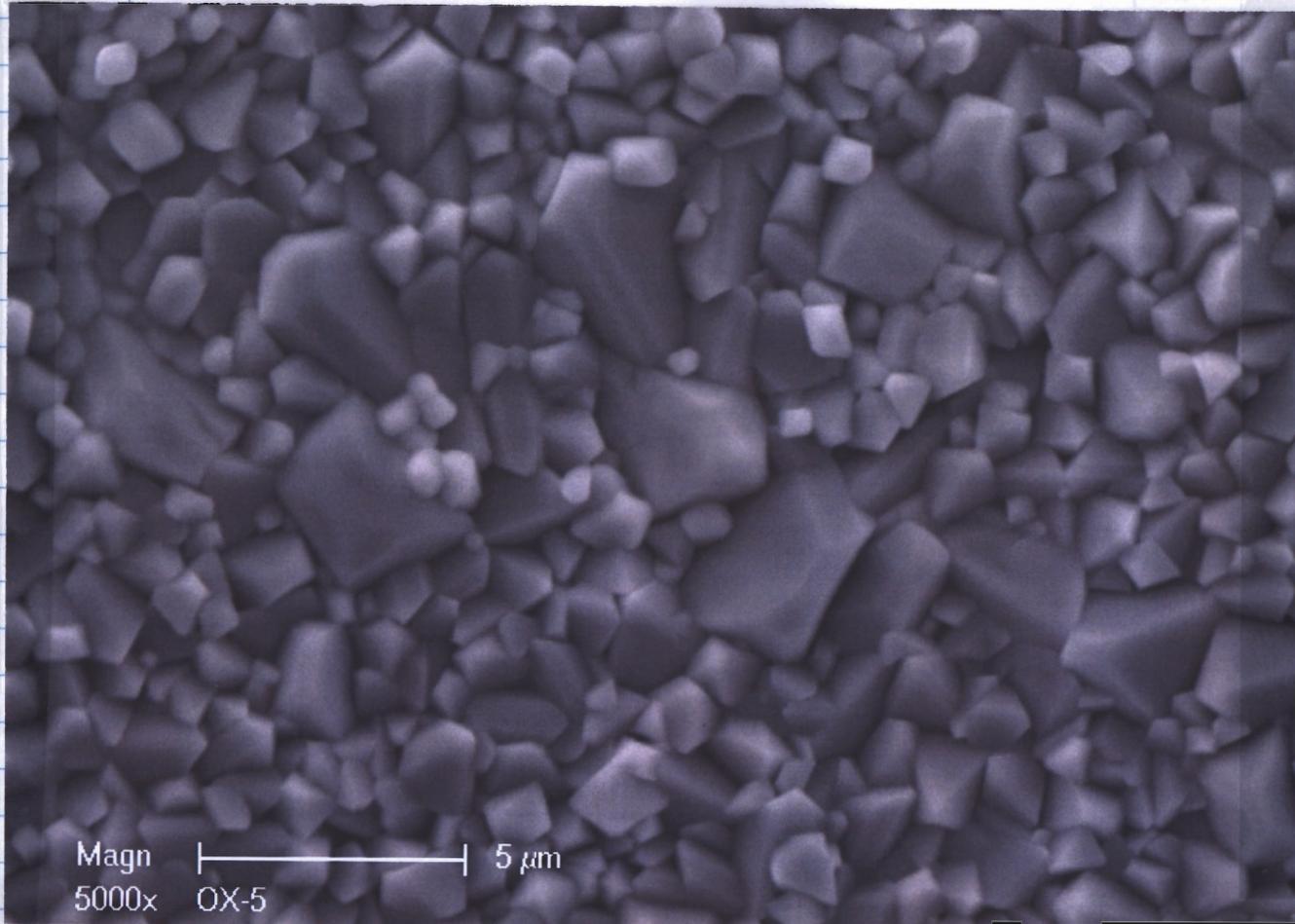
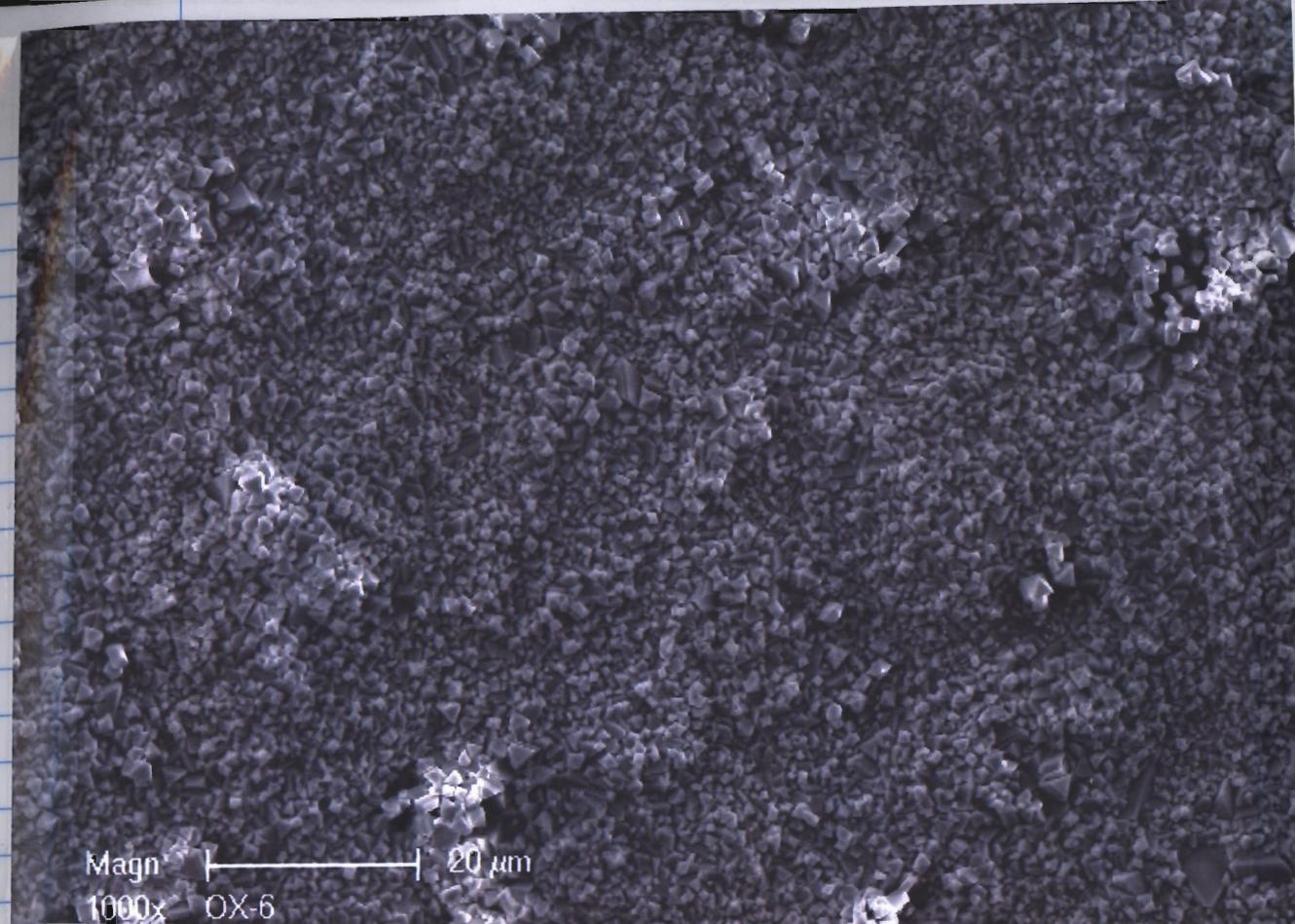
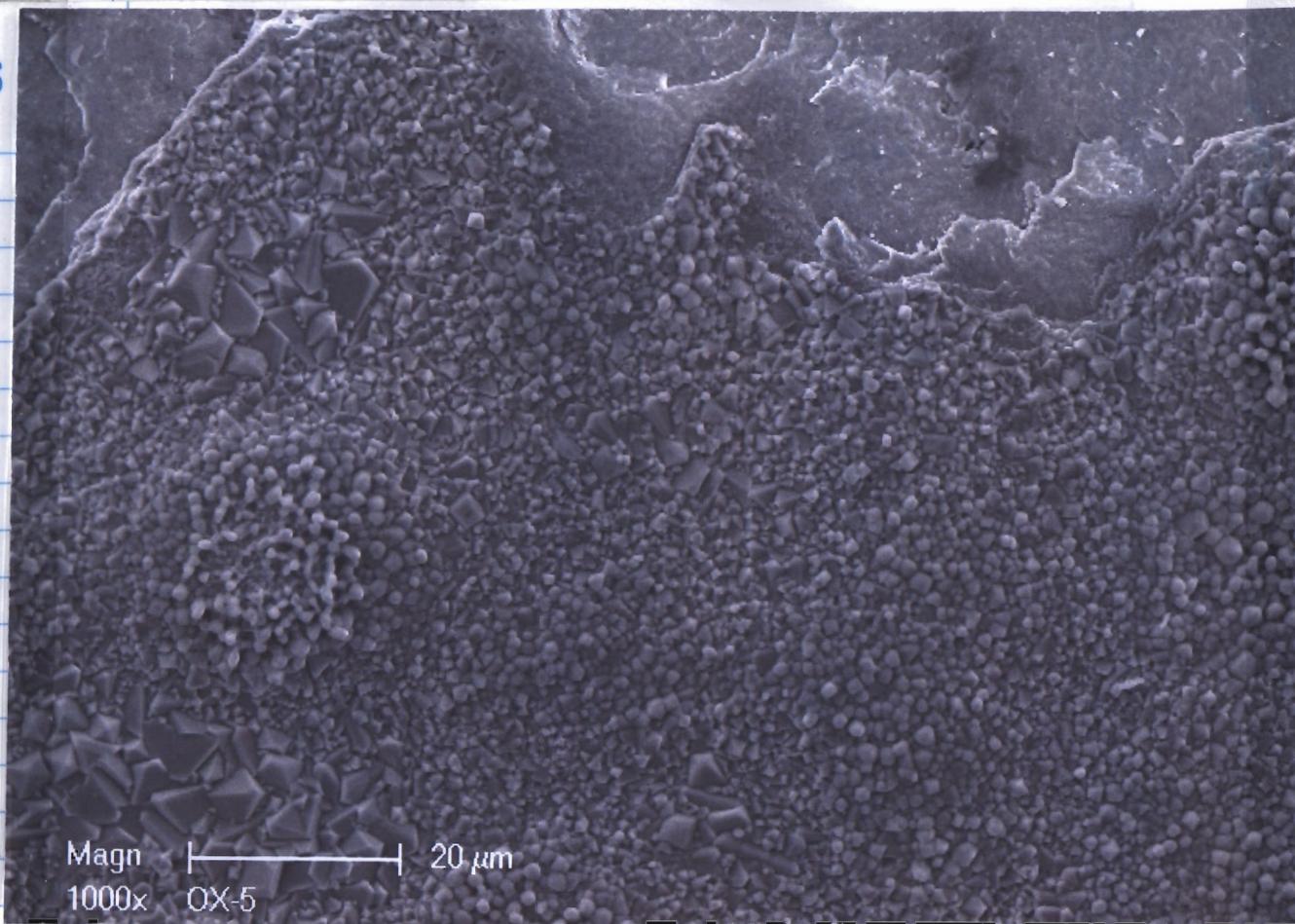


Magn |-----| 5 μ m
5000x OX 2

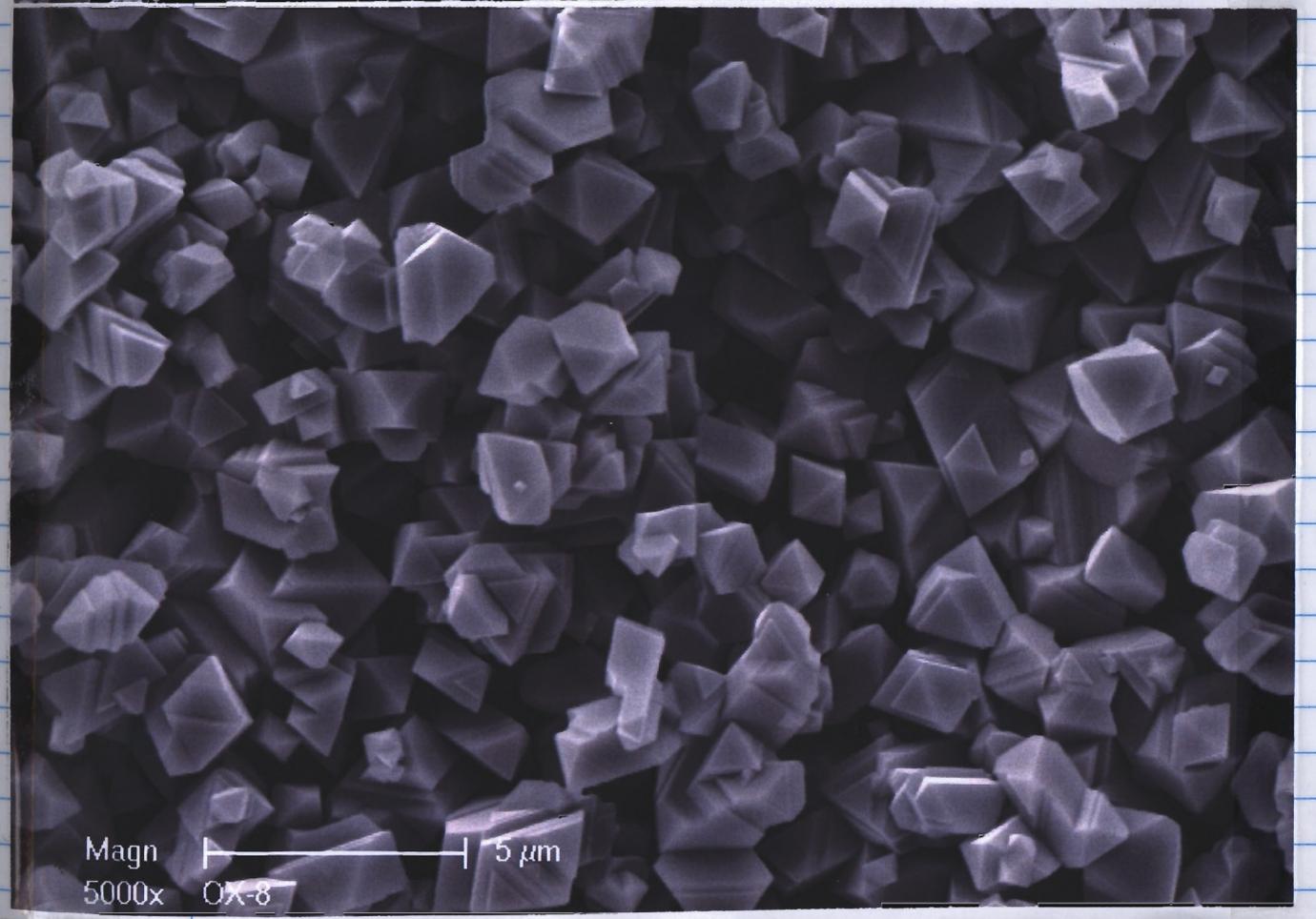
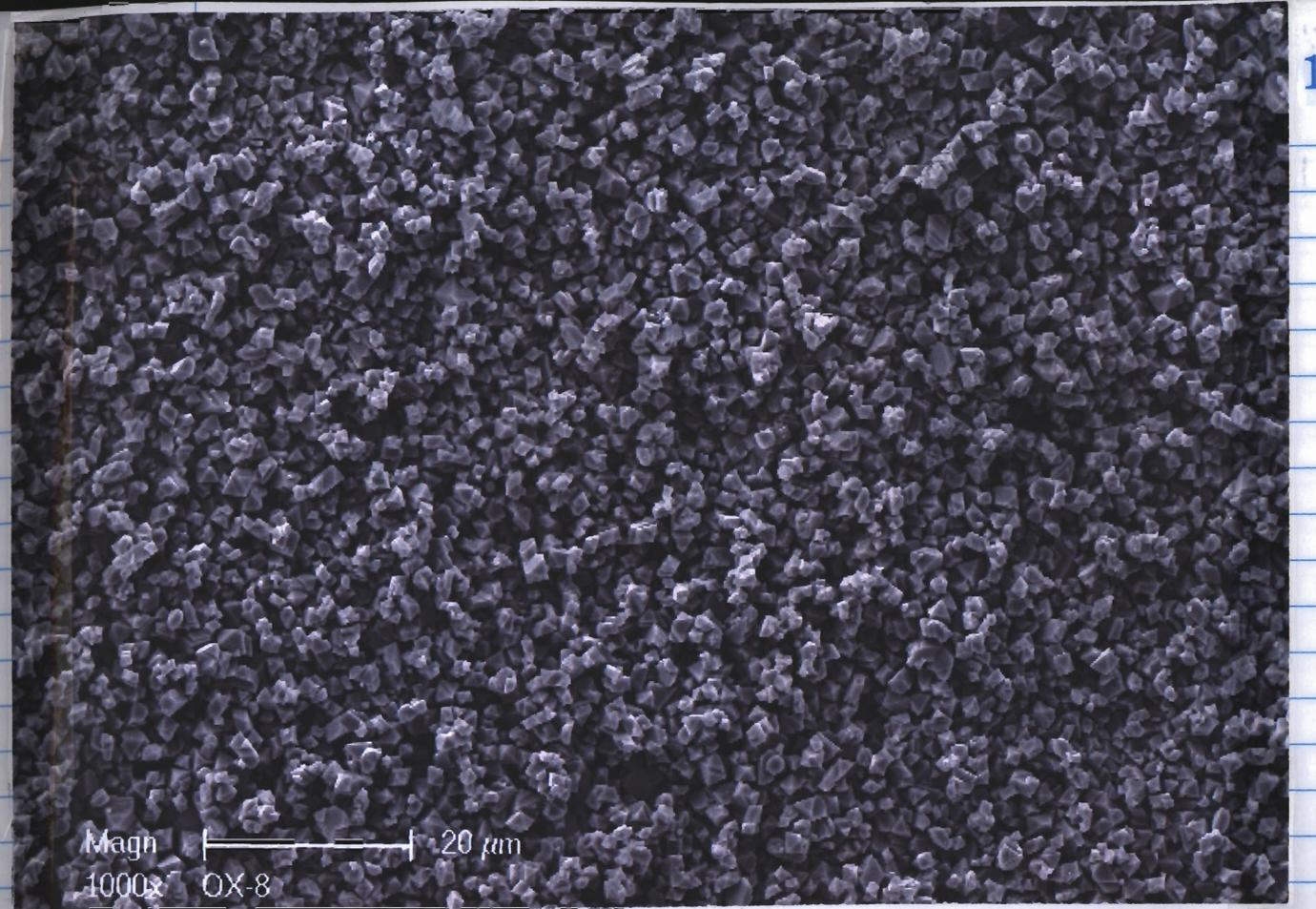
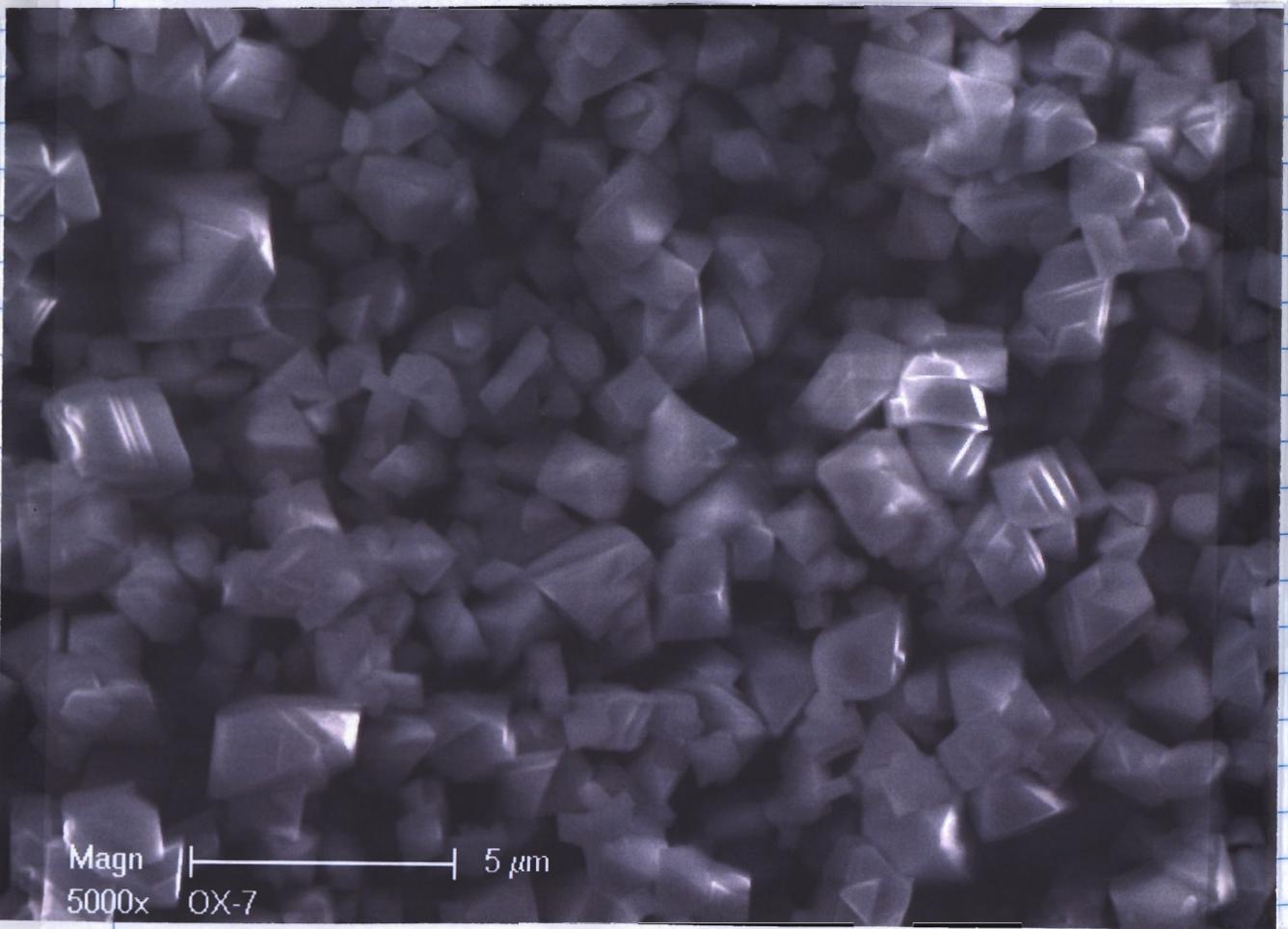
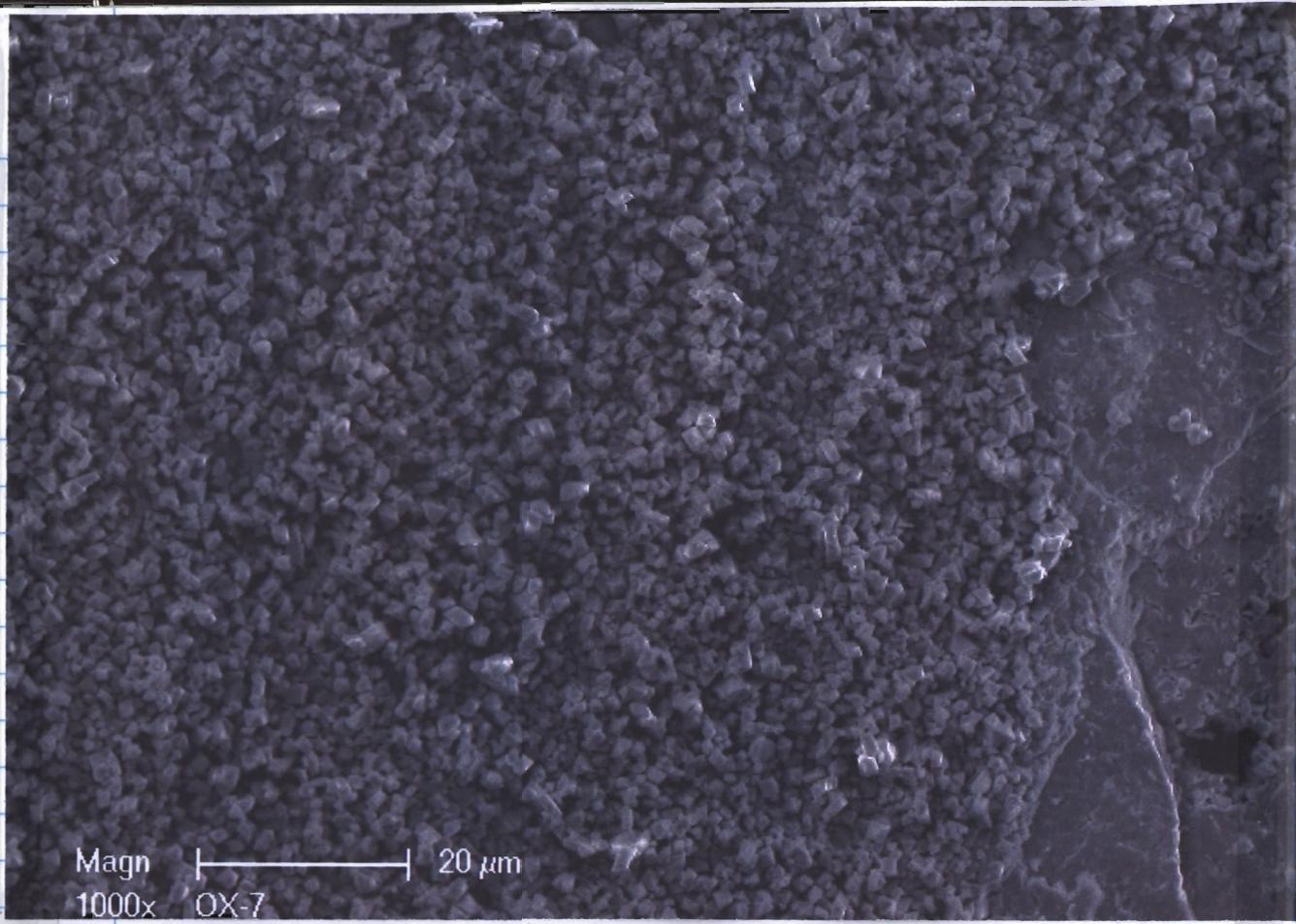
1 February 2007 M.S.



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Task assigned to: Mark Silver

Background:

So that alloy coupons may be examined in cross section by way of scanning electron and reflecting light microscopy, the samples must be mounted in such a way as to allow for the proper exposure of the coupons in a convenient orientation. The coupons are cut in half and one of these halves is mounted in epoxy with the cut exposed at the base of the mount.

Objective: Create epoxy mounts of test specimens

Additional equipment/Materials:

- Struers Epofix cold epoxy mounting kit
- Release agent
- Desiccator
- Leeson SD-90 vacuum pump
1725 RPM
Model # : A6C17DC19A
sn: 79079
- Blue M Single Wall Transite oven
Model # : SW-17TA
sn: S3-7862
- Metal specimen holders ("curl clips")
- Protective mount caps
- Etch pen
- Struers Rotoforce-4 polishing arm with Lupo lubricant attachment
sn: 5250193
- Stuers Rotopol-22 automatic polisher
sn: 5160136
- Stuers DP Suspension, P
6 μ m, 3 μ m, and 1 μ m diamond polishing liquid
- Blue lubricant
- Red lubricant
- Ney ULTRASONIK cleaner
- Isopropyl alcohol in a spray bottle
- Buehler Torramet specimen dryer
115 volt
Cat # : 75-6000-160
sn: 340-TD-350

Procedure followed:

1. Initial mount:

The inner surfaces of the plastic epoxy molds are coated with release agent. A relatively small amount of epoxy is mixed from proportions specified in the instruction booklet for the cold epoxy mount kit and poured into each mold so that only the bottom surface of the mold is covered. The coupons are placed in the center of the epoxy laying flat. Additional epoxy is poured to fill each mold so that the coupons are barely submerged. The mounts are then vacuum impregnated for 15 minutes in order to remove air bubbles. The mounts are then placed in the oven at its lowest temperature setting (just over 100°C) for at least 4 hours to cure the epoxy.

2. Cross-section:

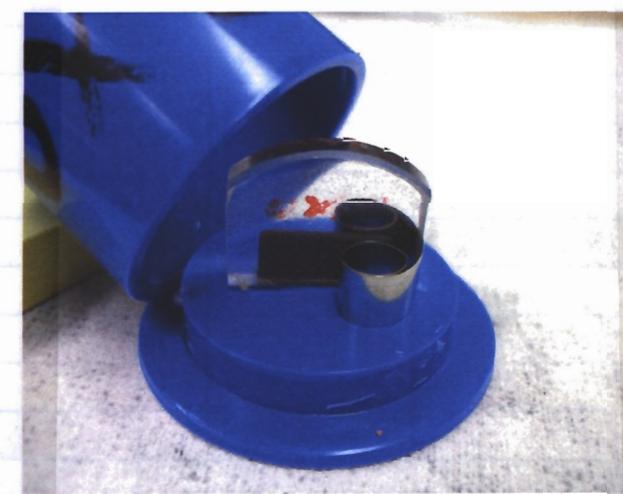
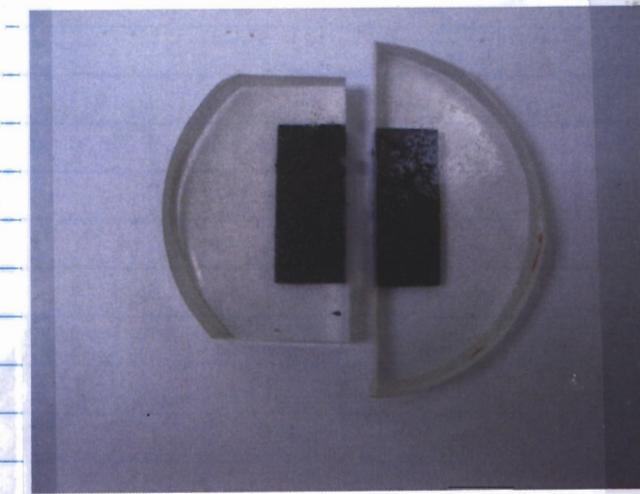
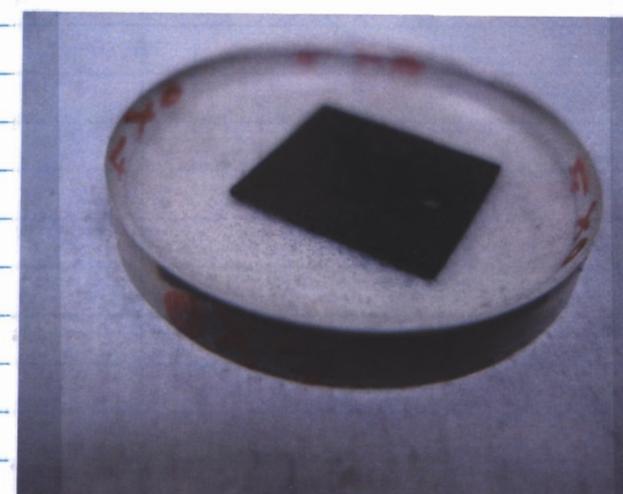
The edges of the epoxy discs created in step one are ground away with the variable speed grinder/polisher (see page 2 for equipment). The epoxy discs are then cut so that the coupons are sectioned in half parallel to the length of the coupons using the precision wet cut-off saw and the rounded edges whose tangents are perpendicular to the length of the coupons are flattened with the grinder.

3. Second mount:

The resulting cross-sections from step 2 are then placed with the exposed cuts face down within the epoxy molds and held upright with curling specimen clips after release agent has been applied to the inner surfaces of the molds. The epoxy is then mixed as in step 1 and then poured over each sample to nearly fill the molds (and to completely submerge the cross-sections). These are then vacuum impregnated for 15 minutes within the desiccator to remove air bubbles and then allowed to cure for at least 4 hours in the oven at the lowest temperature setting.

4. Polishing:

The edges of the mounts are then ground away in the same way as before and then the bottom surfaces containing the exposed cuts of the alloy coupons are uniformly polished to 600 grit on the variable speed grinder/polisher. The mounts are then placed on the Rotopol-22 automatic polisher after the polishing cloth is sprayed with blue lubricant and 6 μ m diamond polishing liquid. The polisher is set to run with a 35 Newton force from the Rotoforce-4 polishing arm for 5 minutes. During the 5 minutes of polishing, the polishing cloth is sprayed with lubricant and polishing liquid every 15 seconds. The mounts are then cleaned in the ultrasonic cleaner, sprayed with isopropyl alcohol and dried under the specimen dryer. The mounts are then polished to with the 3 μ m spray in the same fashion, but using the red lubricant, and then cleaned in the same fashion. The process is repeated with the 1 μ m diamond spray with the blue lubricant, samples are cleaned, capped on the bottom side with the protective mount caps, and the sample ID is etched upon the top side of the mount with an etch pen.



5 February 2007 M.S.

1 March 2007 M.S.

Depth of Oxygen Attack in Coupons

Task assigned to: Mark Silver

Background:

One observation relevant to the degree of oxidation and corrosion of an alloy is how deep into the alloy that oxygen gas can penetrate. The depth of oxygen penetration, or attack, can be measured from magnified images of an alloy cross section.

Objective: Measure and record the depth of oxygen attack in coupons of Alloy C-22 after being exposed to high temperatures.

Additional equipment/Materials:

- Polished epoxy mounts of samples OX1-OX8
- Olympus PMG3 reflecting light microscope
sn: 119835
- Sony DFW-SX900 Photographic equipment
- Clemex Vision PE 3.5 imaging software

Procedure:

Use the reflecting light microscope and associated imaging hardware/software to measure the distance from the edge of each coupon to the furthest interior oxygen bubble of that coupon. This distance is the maximum depth of oxygen attack for that sample.

1 March 2007 M.S.

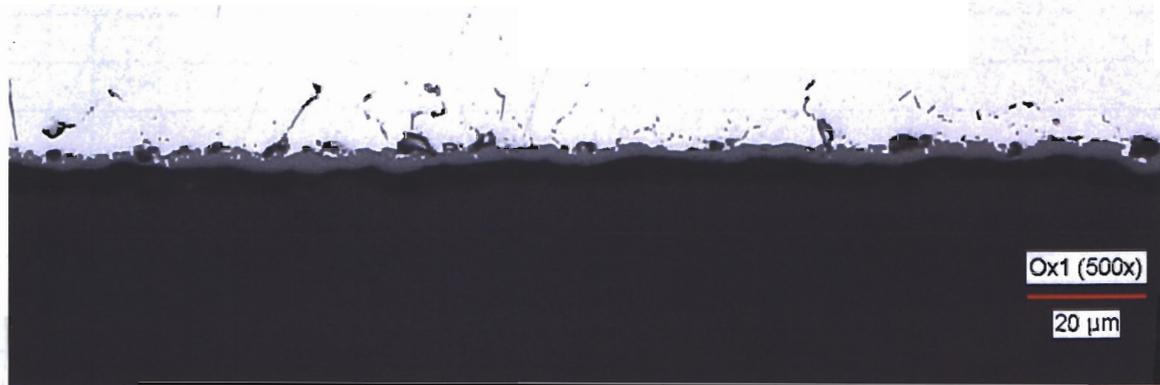
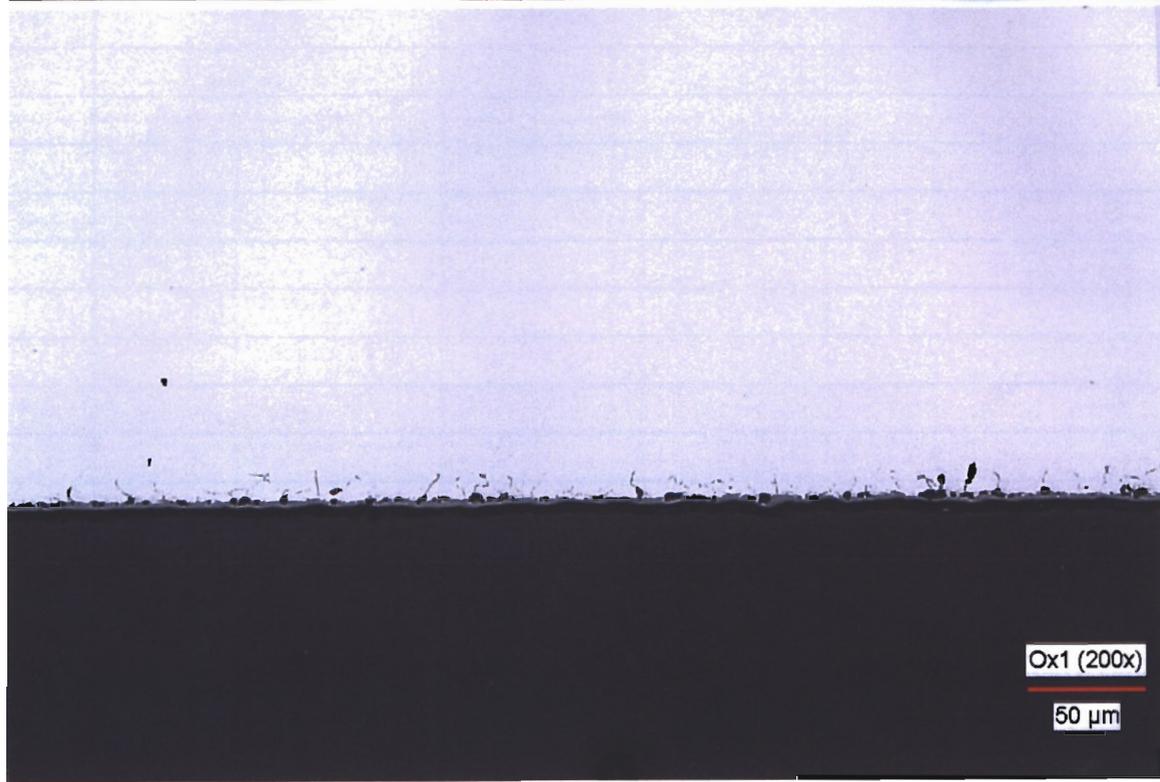
5 March 2007 M.S.

Maximum Depth Measurement of Oxygen Attack:

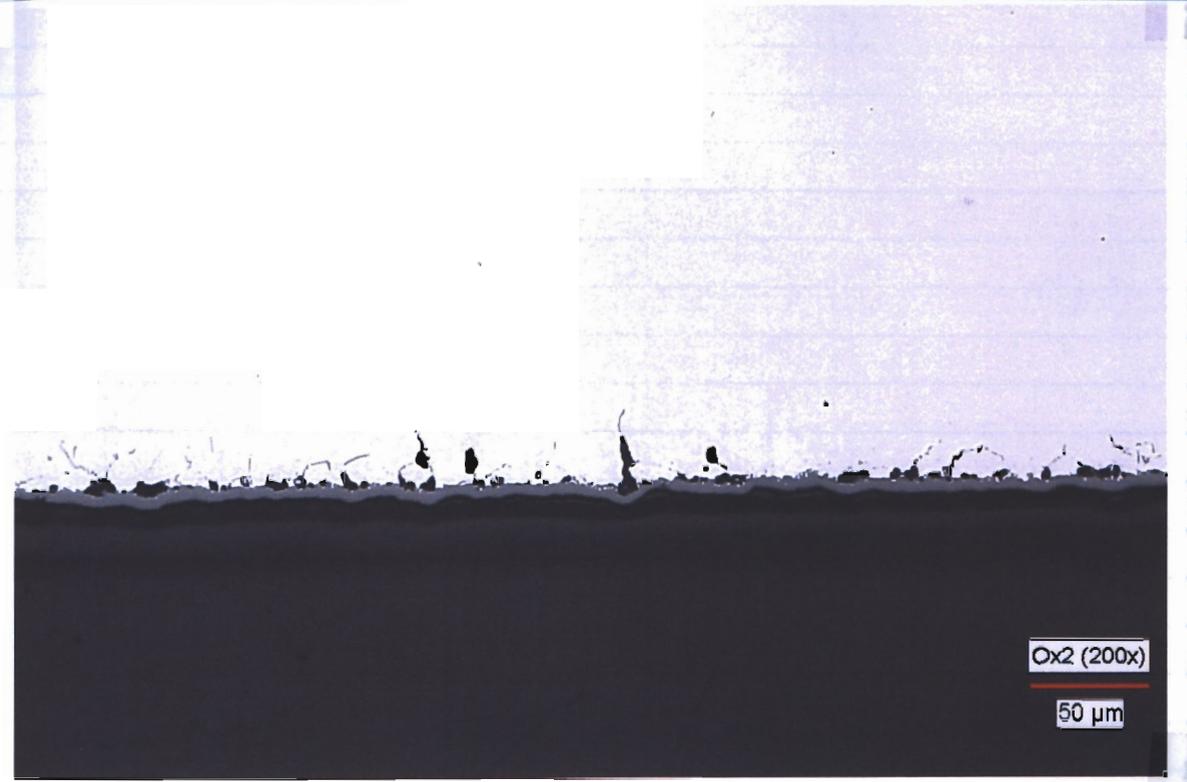
Sample	Measured Depth	External oxide Scale Thickness
<u>OX1</u>	<u>19.84 μm</u>	3.93 μm
<u>OX2</u>	<u>36.89 μm</u>	5.41 μm
<u>OX3</u>	<u>∞</u>	∞
<u>OX4</u>	<u>∞</u>	∞
<u>OX5</u>	<u>45.25 μm</u>	9.67 μm
<u>OX6</u>	<u>50.82 μm</u>	9.84 μm
<u>OX7</u>	<u>105.60 μm</u>	13.77 μm
<u>OX8</u>	<u>130.80 μm</u>	19.18 μm

5 March 2007 M.S.

K. T. Chiril 3/29/07

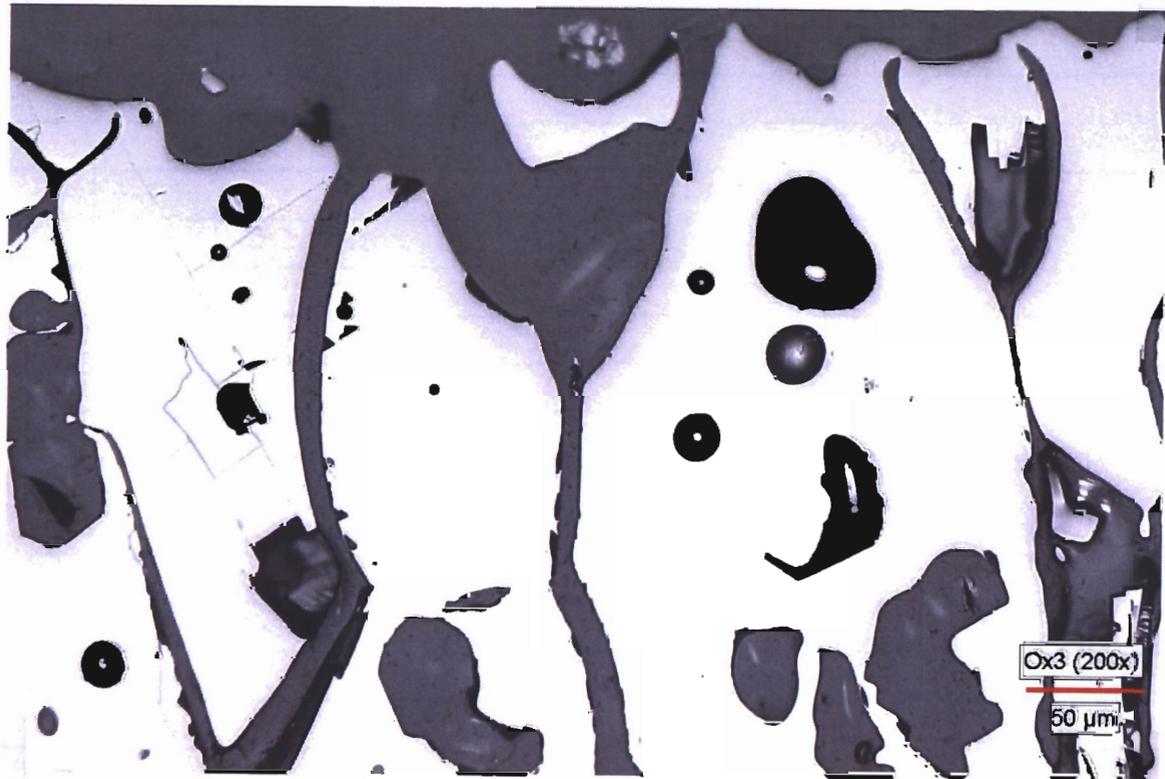
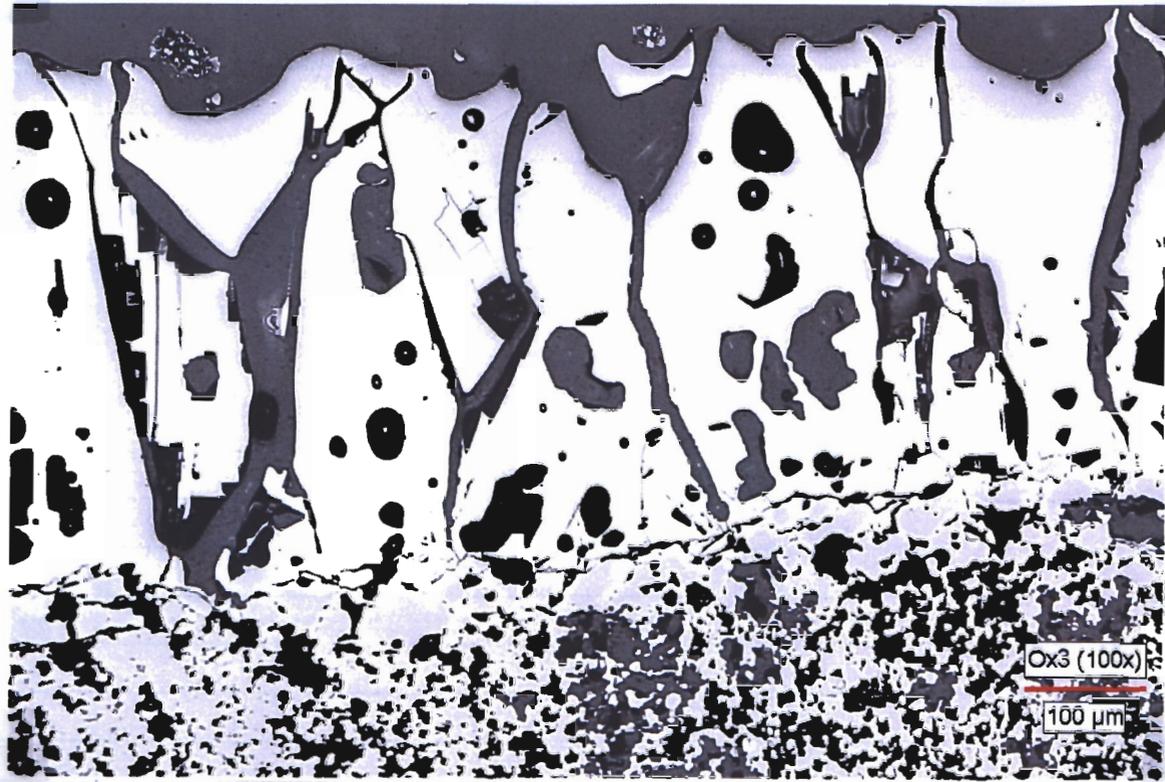


K-T. Chung 3/29/07

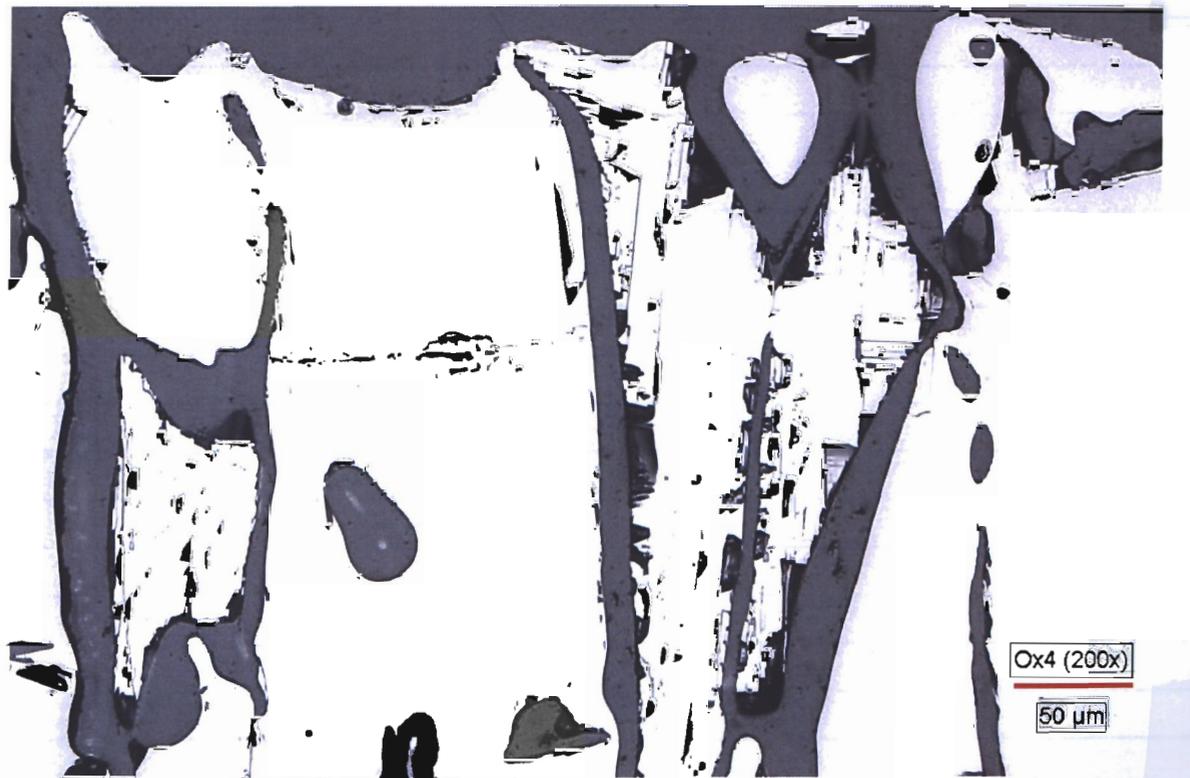
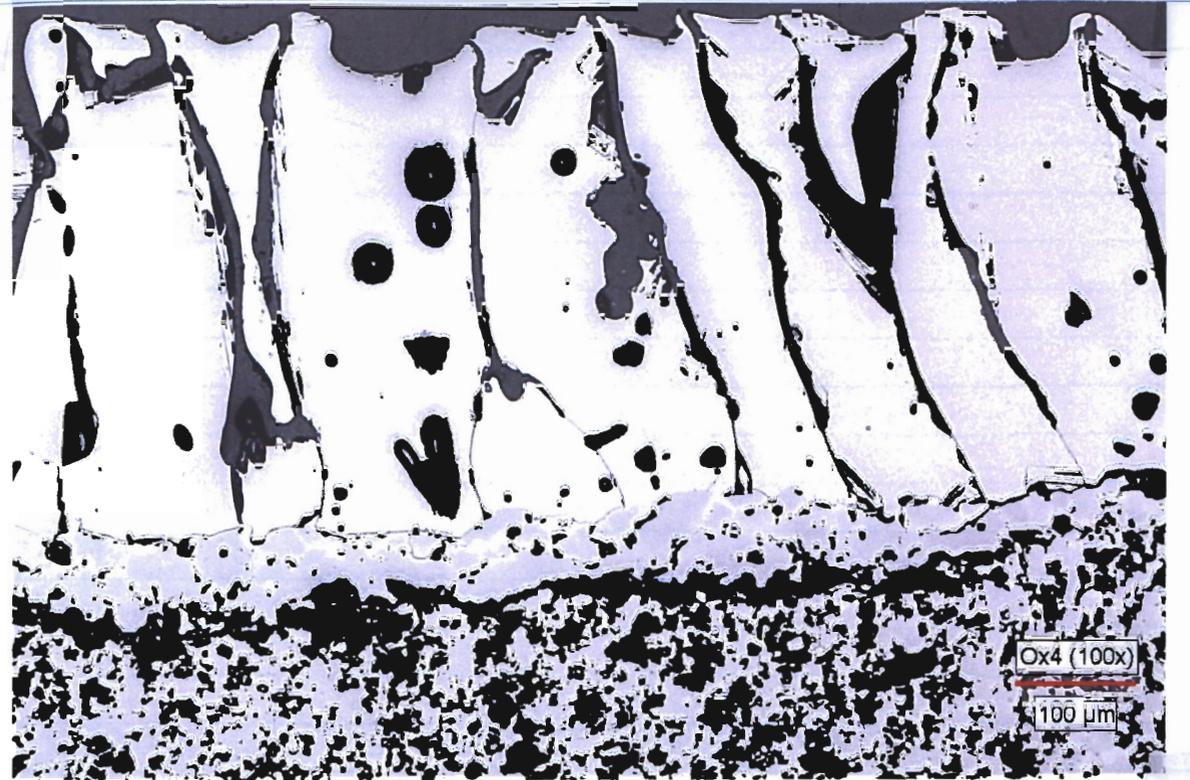


K-T. Chung 3/29/07

5 March 2007 M.R.

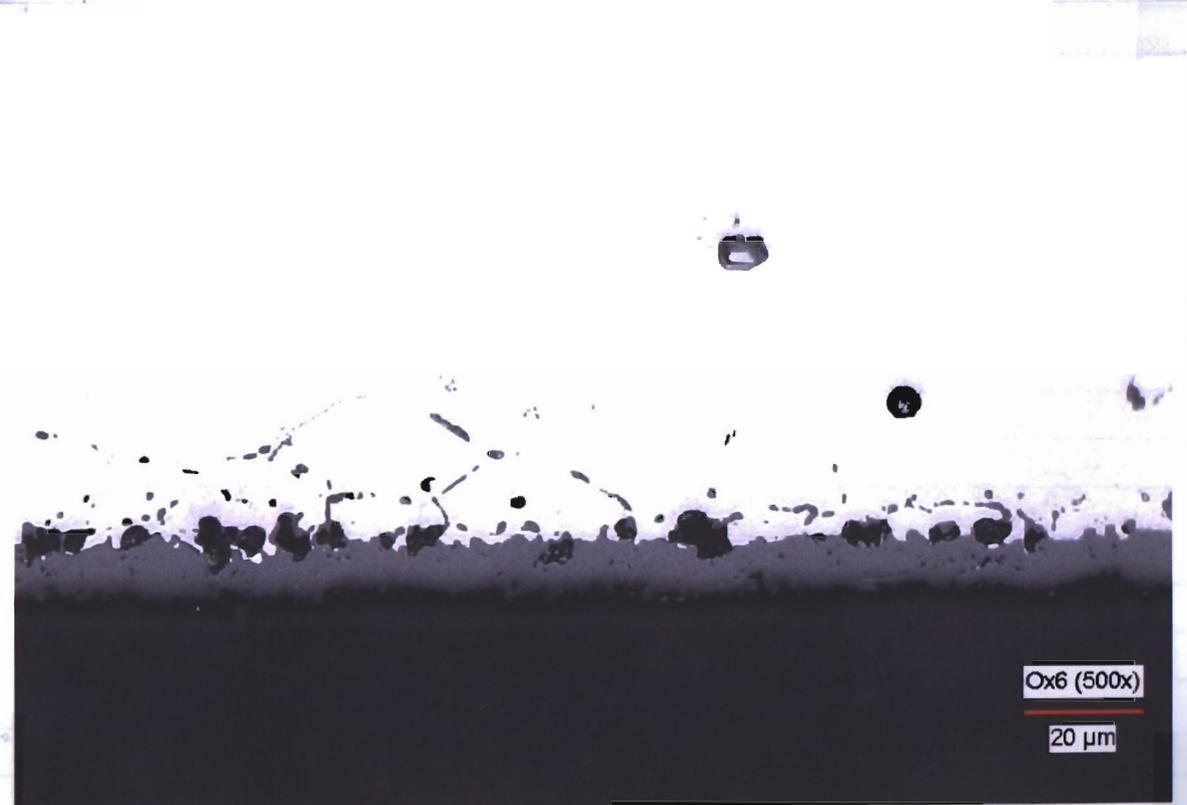
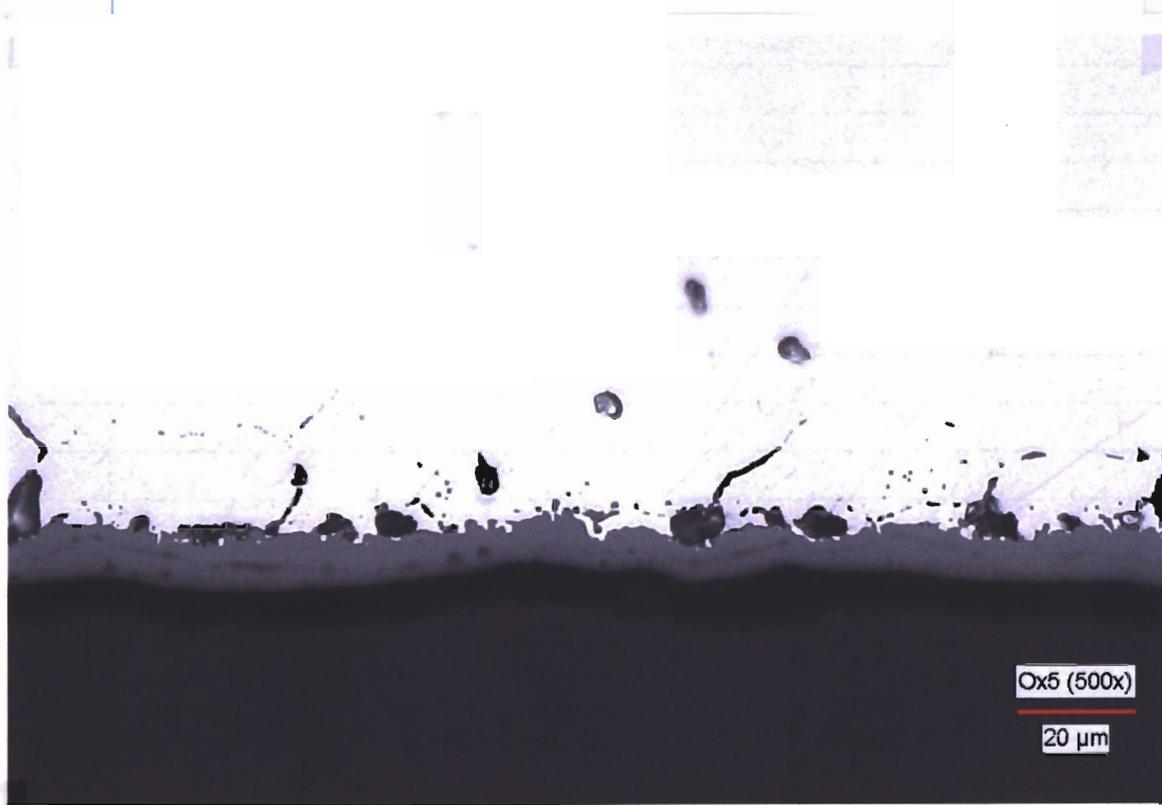
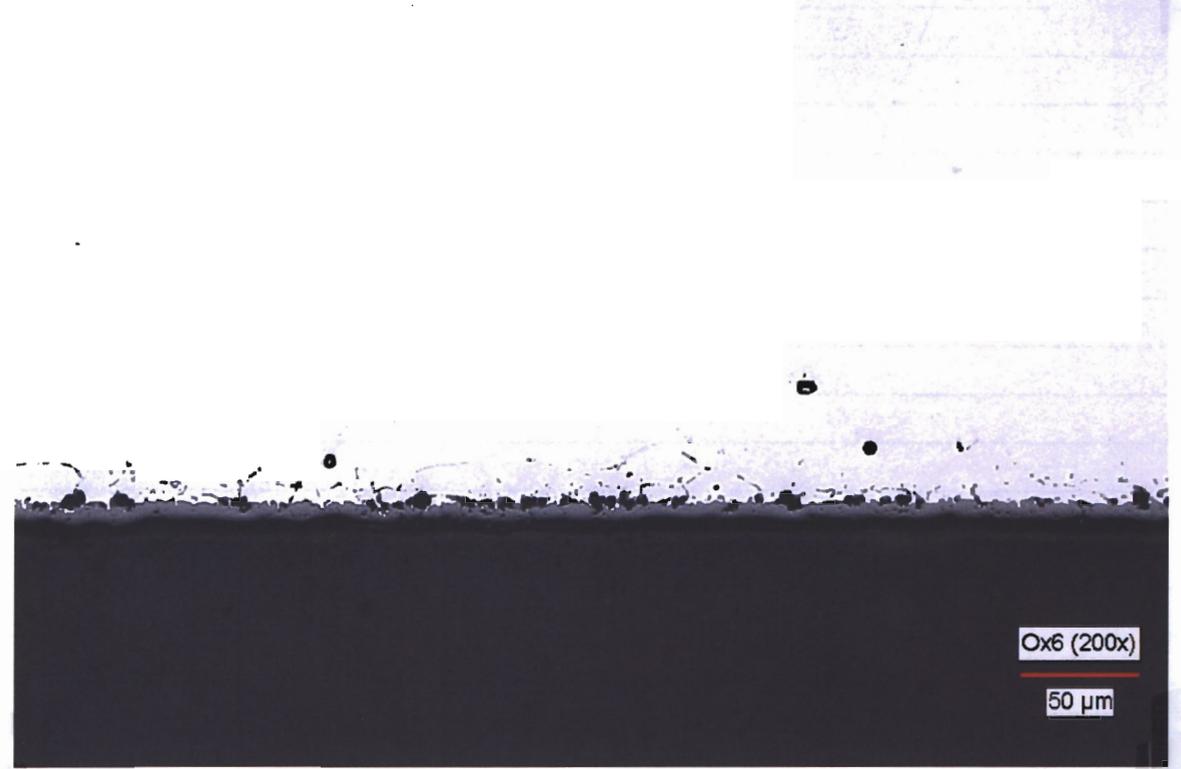
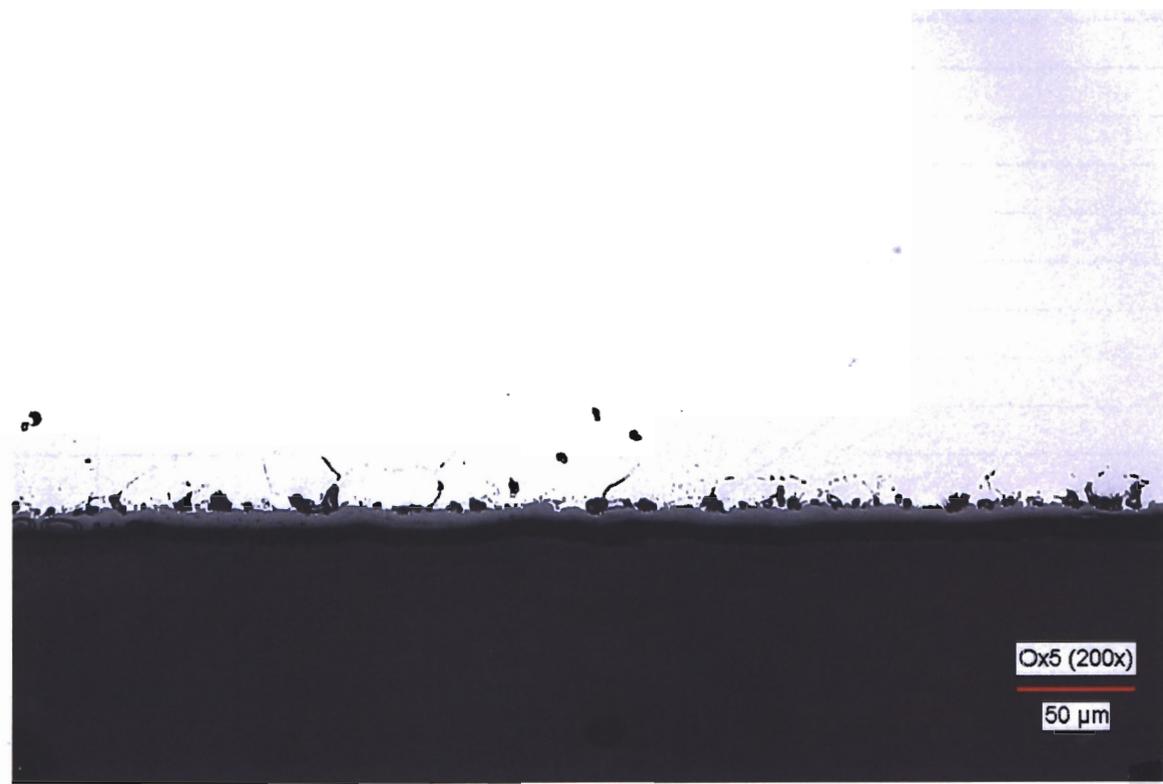


K. T. Chiang 3/29/07



K. T. Chiang 3/29/07

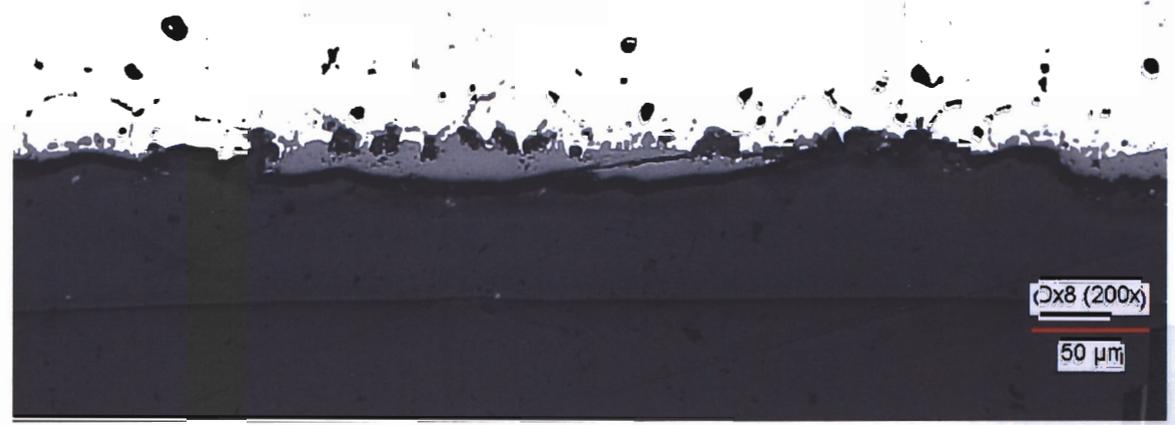
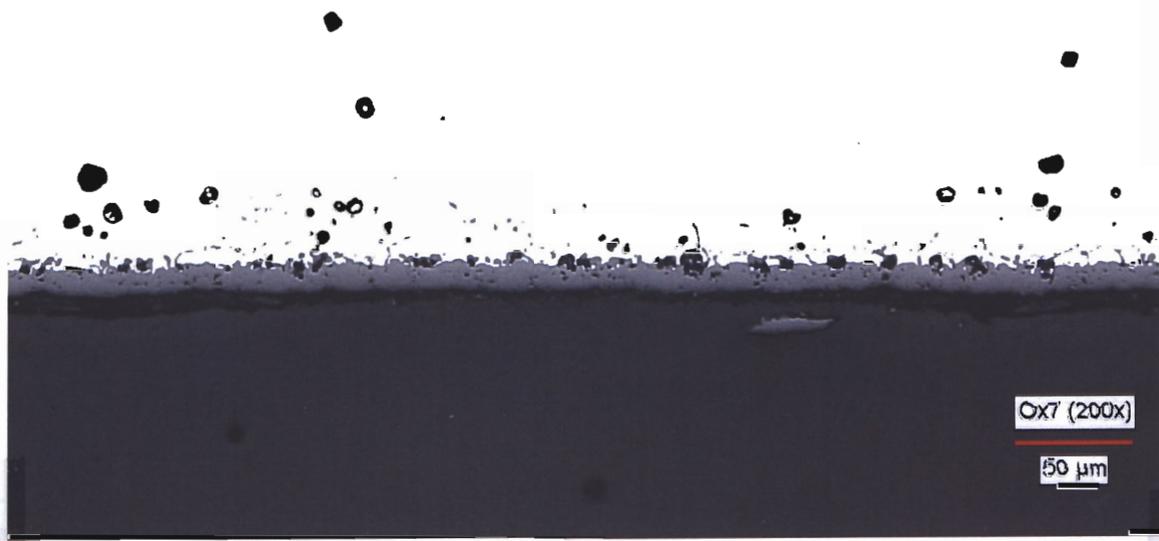
5 March 2007 M.S.



K. T. Ching 3/29/07

K. T. Ching 3/29/07

5 March 2007 M.R.



K. J. Ching 3/29/07

K. J. Ching 3/29/07

K. J. Ching 3/29/07

27 June 2007 M.P.

Summary of Physical Results from Thermal Aging

Sample #	Surface Area (cm ²)	Initial Mass (mg)	Final (mg)		
			Mass of Specimen	Mass of Spall	Total Mass
OX 1	4.443	2359	2329	0	2329
OX 2	4.497	2326	2362	0	2362
OX 3	4.504	2262	643	1939	2582
OX 4	4.52	2398	2689	7	2696
OX 5	4.278	2242	2247	0	2247
OX 6	4.365	2158	2161	0	2161
OX 7	4.405	2257	2232	38	2270
OX 8	4.491	2372	2294	105	2399

This information was prepared by Mark Silver but not compiled and inserted into this notebook. Thus, I've made the entry of Mark's data.

27 June 2007 M.P.

26 April 2007 M.S.

Interaction Between an Alloy Crucible and Contained Magma

Task assigned to: Mark Silver

Objective: Monitor the extent of oxidation and corrosion of alloy C-22 in crucible form when molten rock is heated within it.

Additional equipment/materials:

- Hastelloy C-22 crucible (sample MC-1)
Heat #: 2277-5-3299
Major element composition: 58% Ni, 22% Cr, 13% Mo, 4% Fe, 3% W (estimated by mass)
Machined to a length of 1.5", diameter of 1", with a 1.0" deep bore down the center of the length with a diameter of 0.75"
- Lathrop Wells 1" basalt core (sample LW4-3A)
- Zeiss Stemi SV11 stereo microscope

Procedure:

1. Preparation:

The alloy crucible is cleaned in the ultrasonic cleaner and with acetone. Sample LW4-3A is broken into smaller fragments and the crucible is almost completely filled with the fragments.

2. Heating:

The crucible is incrementally heated in the furnace until the melting point of the basalt fragments is reached. Once the magma is generated, it is then further heated to some specified temperature.

3. Crucible analysis:

Images are taken with photographic equipment attached to the stereo microscope documenting the appearance of all surfaces of the remaining crucible. What solid portion of the crucible remains is then cut in half with the precision wet cut-off saw perpendicular to the length. A cross section is then cut through the length of one half of the crucible and mounted in epoxy for further analysis.

K. J. Chief 4/27/07

Temperature and Duration Specifications for MC-1:

Furnace setpoint: 1000 °C
 Furnace temperature: 1017.5 °C
 Duration of exposure: ≈ 2/3 hrs.
 Placed in furnace: 4:03 pm 02/14/07
 Removed from furnace: 4:44 pm 02/14/07
 Appearance: *Crucible surface Tarnished after heating.*

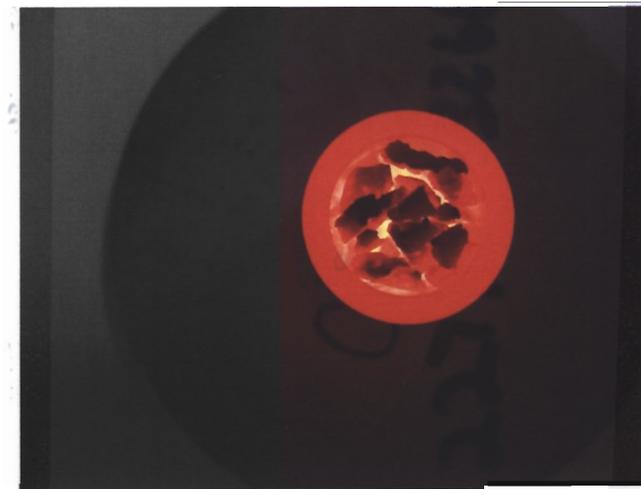


Furnace setpoint: 1040 °C
 Furnace temperature: 1053.5 °C
 Duration of exposure: ≈ 1 hrs.
 Placed in furnace: 4:44 pm 02/14/07
 Removed from furnace: 5:34 pm 02/14/07
 Appearance: *no change*



*K.S. Chung
4/27/07*

Furnace setpoint: 1060 °C
 Furnace temperature: 1066.5 °C
 Duration of exposure: ≈ 1 hrs.
 Placed in furnace: 5:34 pm 02/14/07
 Removed from furnace: 6:24 pm 02/14/07
 Appearance: *No change*



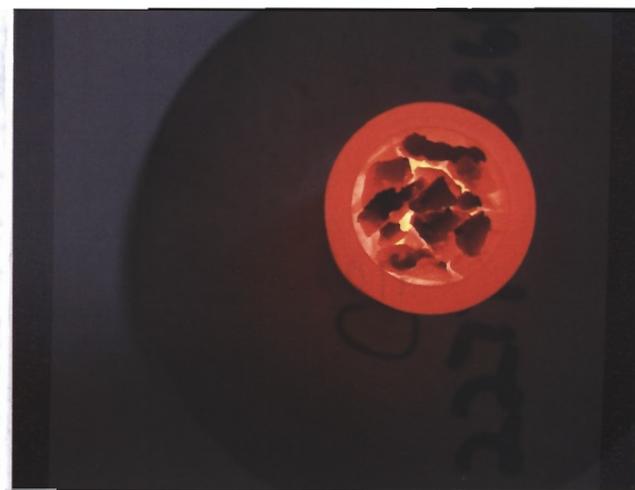
Furnace setpoint: 1080 °C
 Furnace temperature: 1088.9 °C
 Duration of exposure: ≈ 18 hrs.
 Placed in furnace: 6:24 pm 02/14/07
 Removed from furnace: 12:10 pm 02/15/07
 Appearance: *no change*



*K.S. Chung
4/27/07*

26 April 2007 M.S.

Furnace setpoint: 1100 °C
 Furnace temperature: 1109.8 °C
 Duration of exposure: 2 hrs.
 Placed in furnace: 12:10pm 02/15/07
 Removed from furnace: 2:10pm 02/15/07
 Appearance: *No change*



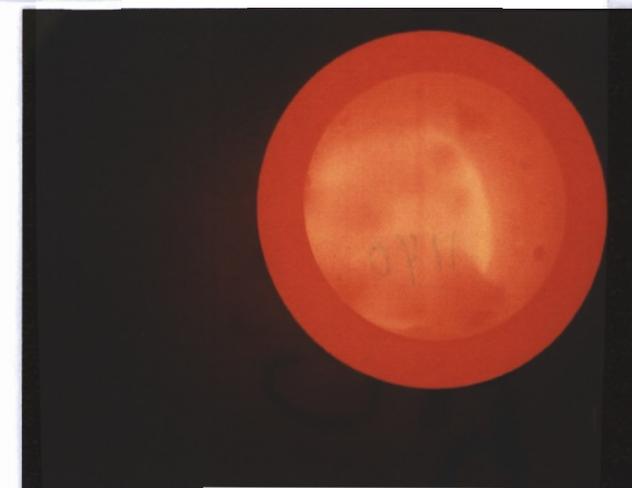
Furnace setpoint: 1120 °C
 Furnace temperature: 1121.9 °C
 Duration of exposure: ≈ 1 hrs.
 Placed in furnace: 2:10pm 02/15/07
 Removed from furnace: 3:12pm 02/15/07



Appearance: *Basalt chips fuse slightly at their edges and appear greasy (close to melting temp.)*

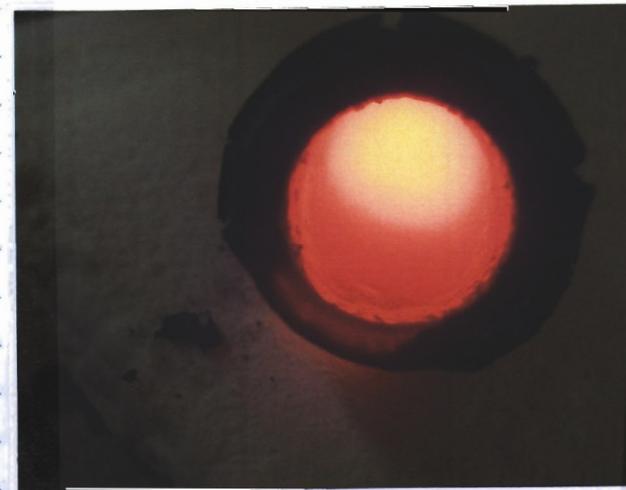
*K.T. Chui
4/27/07*

Furnace setpoint: 1140 °C
 Furnace temperature: 1143.6 °C
 Duration of exposure: ≈ 1 hrs.
 Placed in furnace: 3:12pm 02/15/07
 Removed from furnace: 4:22pm 02/15/07
 Appearance: *Molten*



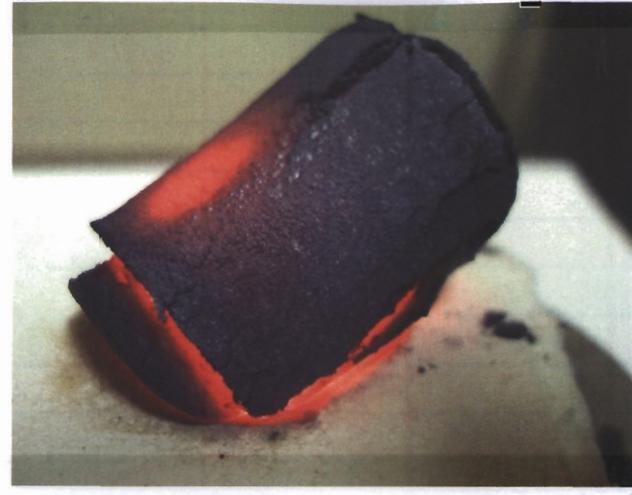
Furnace setpoint: 1255 °C
 Furnace temperature: 1252.6 °C
 Duration of exposure: ≈ 20 hrs.
 Placed in furnace: 4:22pm 02/15/07
 Removed from furnace: 12:20pm 02/16/07

Appearance: *Much of alloy is consumed and material of balls of corroded remains.*



*K.T. Chui
4/27/07*

26 April 2007 M.S.



* Remaining solid portion of alloy surrounded by loose spall. (above and left)

K. J. Ching 4/27/07



Large spall fragment adhering to fibrefrax.

K. J. Ching 4/27/07

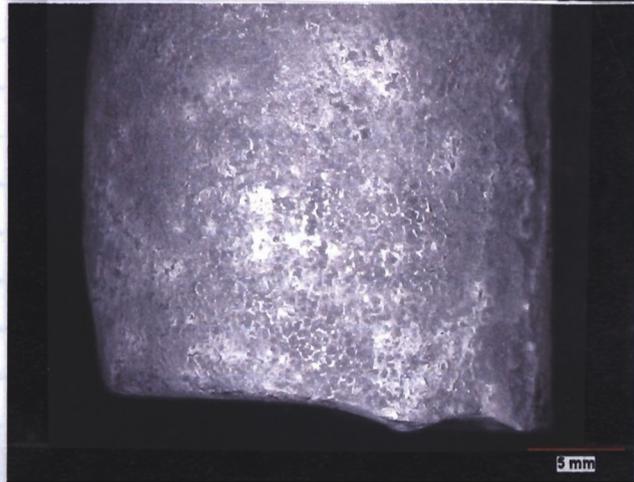
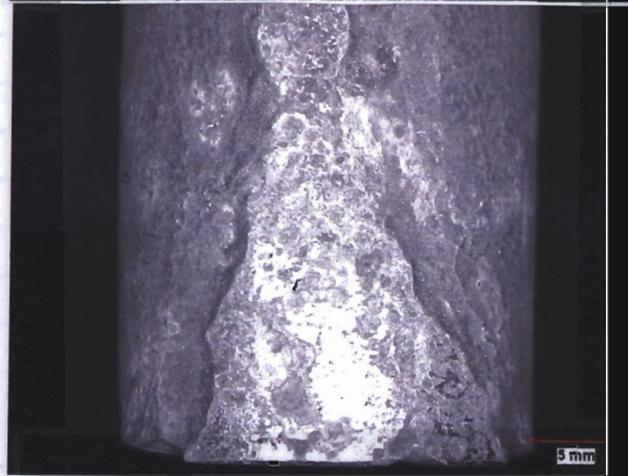
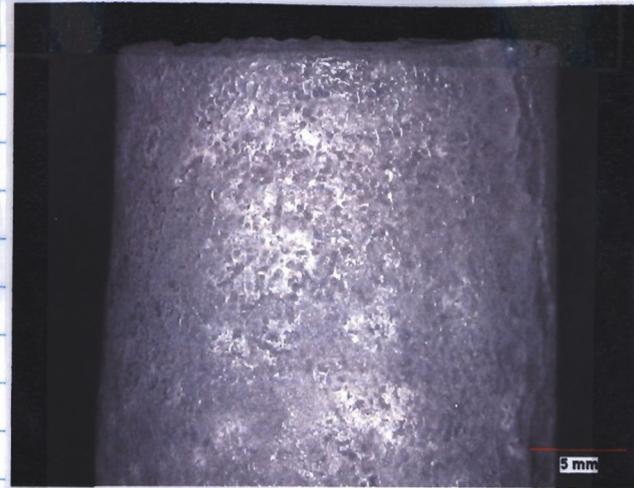
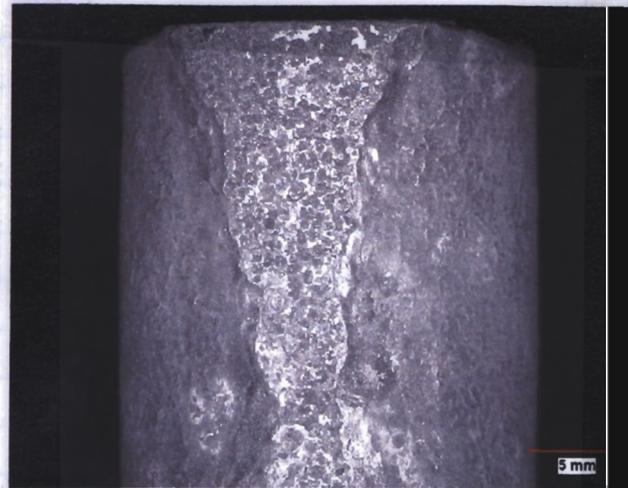


Original Crucible

26 April 2007 M.S.

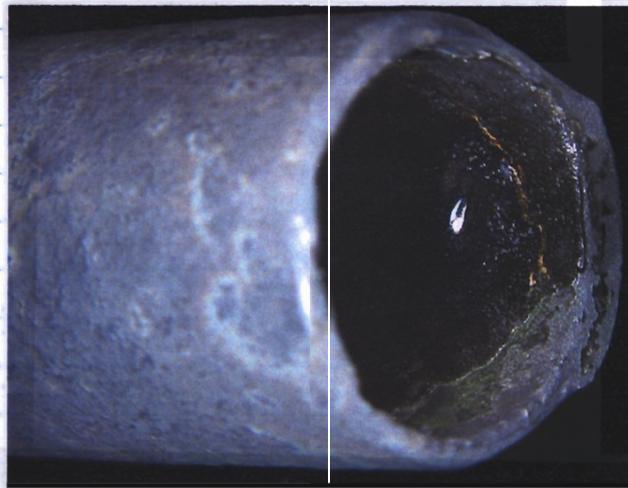
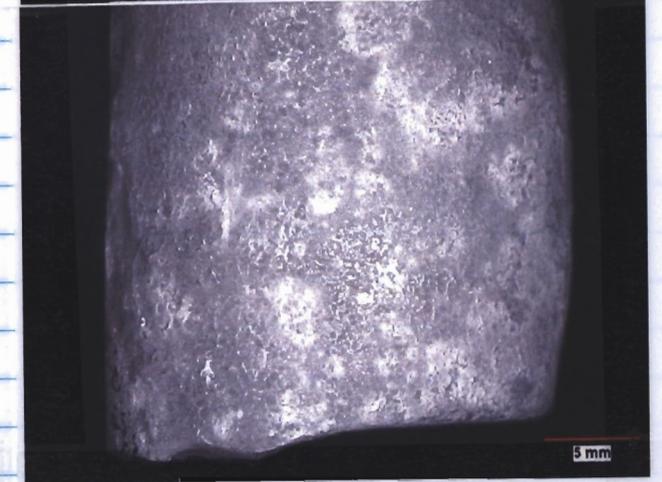
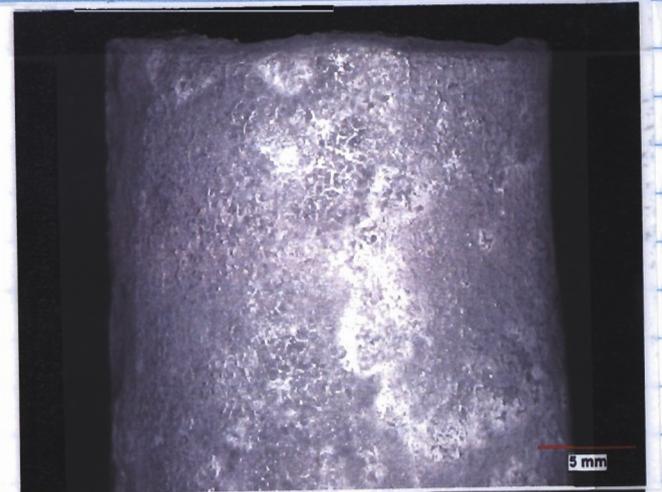
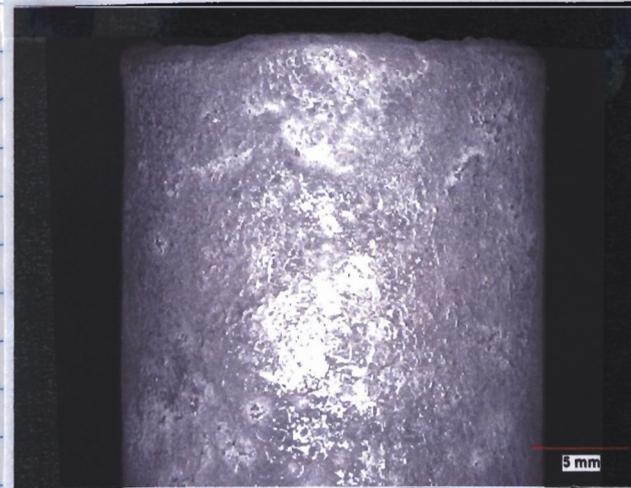
Bottom

Left



Top

Right



* Magnified images of remaining solid portion of alloy / crucible after magma heated to 1252.6°C



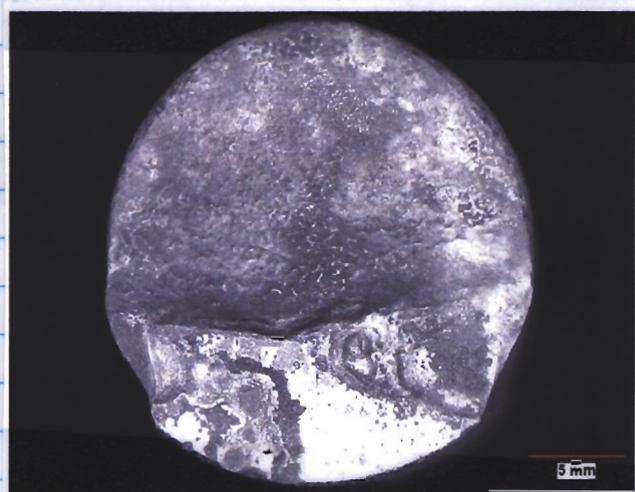
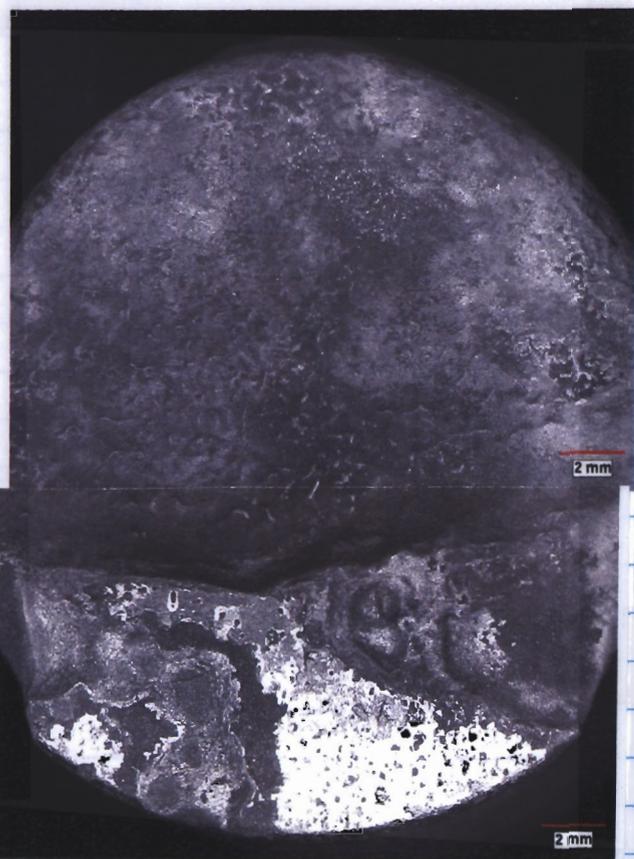
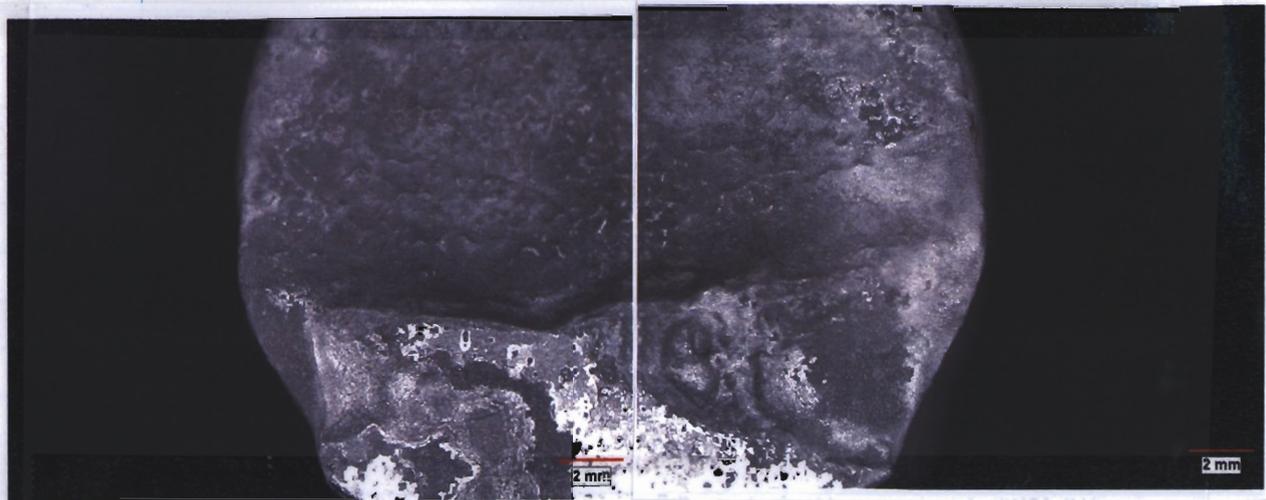
Large spall fragment adhering to Fibrebrax

Interaction interface 26 April 2007 M.S. K.-T. Ching 4/27/07

K.-T. Ching

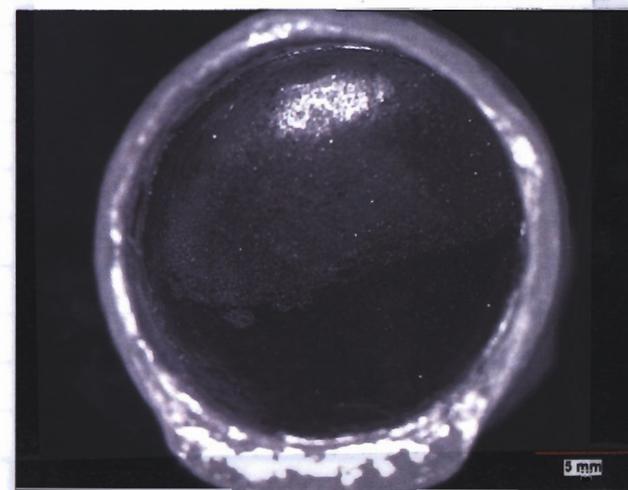
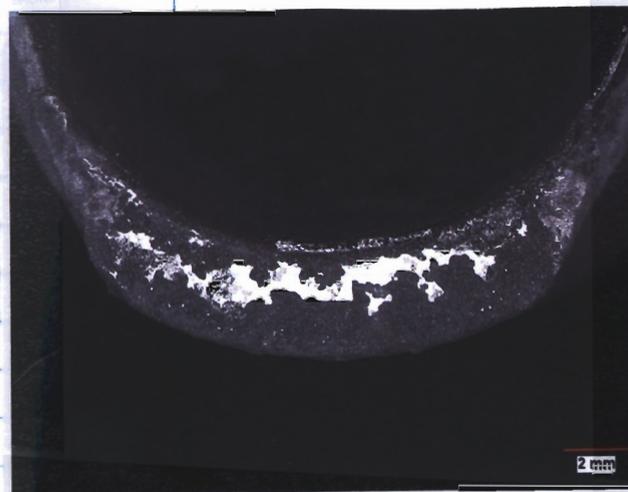
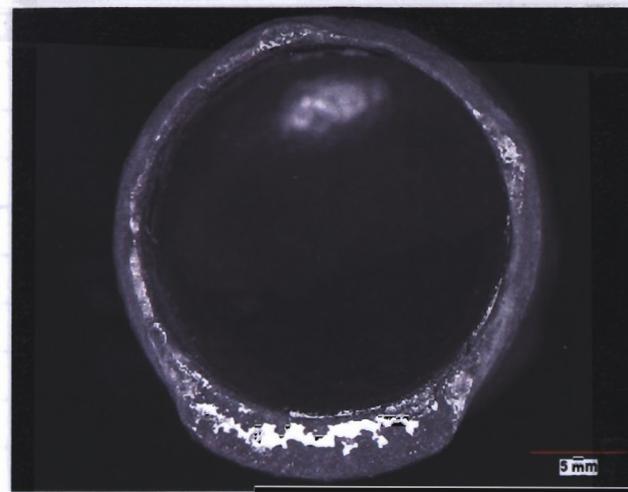
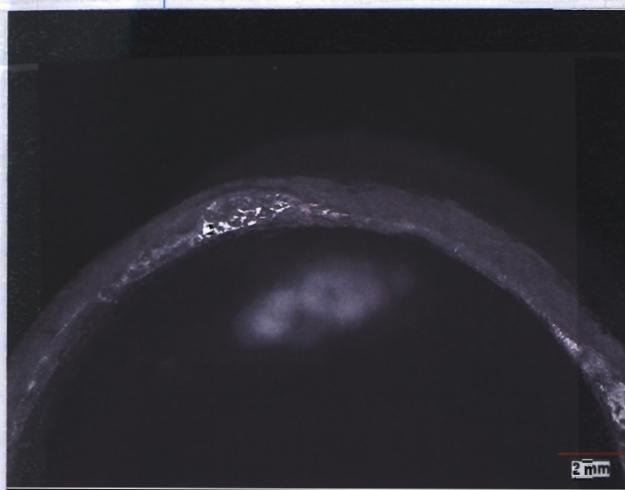
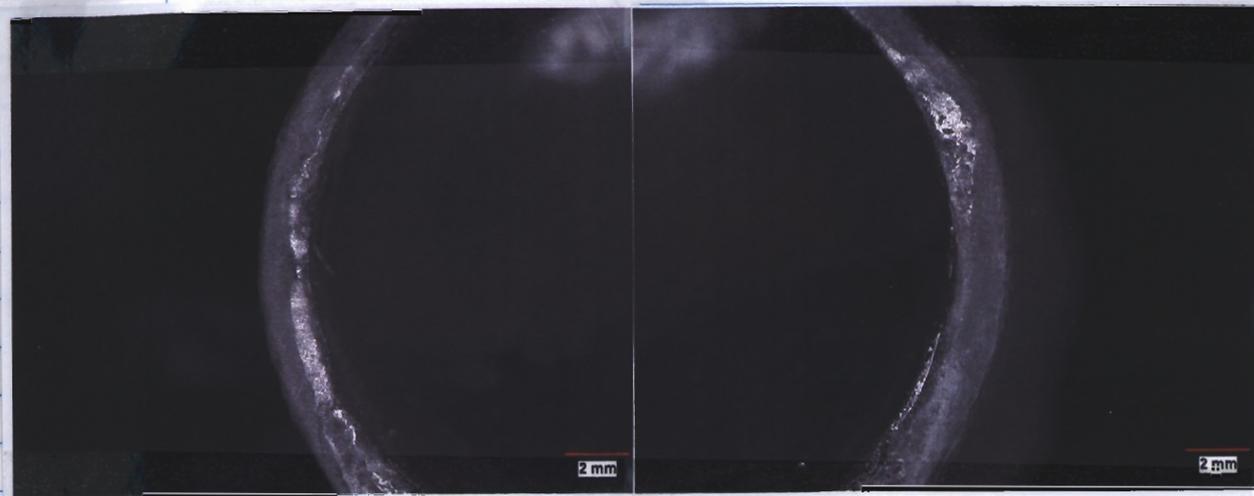
4/27/07

Bottom



H. T. Ching 4/27/07

Top



H. T. Ching 4/27/07

M. S.
26 April 2007

11 June 2007 M.P.

Repeat Experiment (Heat Induced Oxidation & Coating Tests)

Task Assigned to: Matthew J. Panariello

Objective: To further analyze the extent of oxidation and corrosion of Alloy C-22 when coated with slurry and exposed to high temperatures.

Equipment/Materials:

- Hastelloy C-22 coupons
HT#: 2277-3-3266
Major element composition: 58% Ni, 22% Cr, 13% Mo, 4% Fe, 3% W
Machined dimensions (approx.): 1.25cm x 1.44cm x .15cm, .16cm diameter hole .33cm below center of width
- Alumina crucibles, 1" diameter (6)
- 50 mL glass beakers (2)
- Acetone
- Isopropyl Alcohol
- Yucca Mountain rock sample
- Distilled Water
- Platinum wire
- Fisher twist-tie sample bags
- Steel tongs
- Tweezers
- Heat resistant safety gloves
- Safety glasses
- Fisher Scientific FS 14 Ultrasonic Cleaner
- Cold epoxy mounting kit
- 120, 240, 320, 600 grit adhesive backed 8" diameter abrasive discs
- Buehler Ecomet 3 variable speed grinder/polisher
120 volt
490 Max RPM
Cat #: 60-1960-160
sn: 462-A3P-0208
- Buehler Isomet Plus precision wet cut-off saw
115 volt
4500 Max RPM
Cat #: 11280-160
sn: 452-I5P-835
- CM Inc. Rapid Temp. Furnace
Max temp: 1700°C
sn: 029786
Model #: 98002-24
- Lindberg Oven
Max temp: 1200°C
sn: 909172
Model #: 51333
- Starrett Electronic Digital Caliper
Model #: 721
sn: 03031512
Calibration due: Mar 1, 08
- Sartorius Genius Scale (Analytical Balance)
Mass range: .00001g to 200g
sn: 12809099
Calibration due: Nov 11, 07
- Corning Stirrer/Hotplate

K.T. Ching 6/18/07

Identified Sources of Error:

- Mass lost from spalling during heating
- Any difference in chemical or physical composition of different samples
- Any impurities that may have been encountered by the samples between heating and storing

Procedure:

1. Preparation of Alloy C-22 coupons:

The cut and polished specimen are removed from their Fisher Scientific storage bags and weighed on the Sartorius analytical balance. Then, using the digital calipers, the length, width, and thickness of each coupon is measured and recorded. After measuring is complete, the samples are cleaned in two steps. First, each sample is soaked in acetone in a 50 mL beaker and placed in the ultrasonic cleaner for approximately 3 minutes each. Then each sample is rinsed down with isopropyl alcohol and air dried. Finally, the cleaned specimens are suspended in their own alumina crucibles from platinum wires.

2. Oxidizing the Alloy C-22 coupons:

Every sample is at least oxidized by heating it for some period of time in the furnace. Four of the specimens are oxidized at 900°C for 20 hours, one is oxidized at 1200°C for 20 hours, and one is oxidized at 1200°C for 4 hours. After heating, every specimen is allowed to gradually cool down, so as to prevent thermal shock, before reweighing. The gradual cooling process consists of approximately 15 min. in the Lindberg oven at 600°C and another 10 min. on the cooling plate at room temperature (~20°C).

3. Thermal Aging Interaction between Alloy C-22 coupons and Slurry:

Initially, slurry is prepared by mixing some of a Yucca Mt. rock sample (ground into powder) with distilled water. This slurry is then used to coat four of the specimens, which are allowed to dry over a hotplate. The specimen are then suspended in their crucibles from the platinum wire and placed in the furnace at varying temperatures (1050°C to 1200°C) and amounts of time (2 to 20 hours). Again, each specimen is allowed to gradually cool down before it is reweighed.

4. Oxidation and Corrosion Analysis:

The samples are first mounted and polished as cross-sections in epoxy by the method described on pages 20 and 21 in this notebook. Then, using an optical microscope, the cross-sections are photographed at different magnifications and analyzed to determine the external scale thickness and maximum penetration depth of oxygen in each specimen.

11 June 2007 M.P.

K.T. Ching 6/18/07

12 June 2007 M.P.

Sample OX9 Coated with Slurry (Y/N) Mass per unit area _____

Furnace Setpoint: 900 °C
 Furnace Temperature: 902 °C
 Duration of exposure: 20 hrs.
 Start time-date: 12:19 - 6/5/07
 End time-date: 8:19 - 6/6/07

Details/Results:

The sample was oxidized, turning it black

Initial mass: 2.35501 g
 Final mass: 2.35594 g
 Mass of spall: 0 g
 Total mass: 2.35594 g

Sample OX10 Coated with Slurry (Y/N) Mass per unit area _____

Furnace Setpoint: 900 °C
 Furnace Temperature: 902 °C
 Duration of exposure: 20 hrs.
 Start time-date: 12:19 - 6/5/07
 End time-date: 8:19 - 6/6/07

Details/Results:

The sample was oxidized, turning it black

Initial mass: 2.39145 g
 Final mass: 2.39244 g
 Mass of spall: 0 g
 Total mass: 2.39244 g

Sample OX11 Coated with Slurry (Y/N) Mass per unit area _____

Furnace Setpoint: 900 °C
 Furnace Temperature: 902 °C
 Duration of exposure: 20 hrs.
 Start time-date: 12:19 - 6/5/07
 End time-date: 8:19 - 6/6/07

Details/Results:

The sample was oxidized, turning it black

Initial mass: 2.32792 g
 Final mass: 2.32883 g
 Mass of spall: 0 g
 Total mass: 2.32883 g

K. J. Chiang 6/18/07

12 June 2007 M.P.

Sample OX12 Coated with Slurry (Y/N) Mass per unit area _____

Furnace Setpoint: 900 °C
 Furnace Temperature: 902 °C
 Duration of exposure: 20 hrs.
 Start time-date: 12:19 - 6/5/07
 End time-date: 8:19 - 6/6/07

Details/Results:

The sample was oxidized, turning it black

Initial mass: 2.34628 g
 Final mass: 2.34735 g
 Mass of spall: 0 g
 Total mass: 2.34735 g

Sample OX9 Coated with Slurry (Y/N) Mass per unit area .215 mg/mm²

Furnace Setpoint: 1050 °C
 Furnace Temperature: 1051 °C
 Duration of exposure: 2 hrs.
 Start time-date: 9:45 - 6/6/07
 End time-date: 11:45 - 6/6/07

Details/Results:

The powdery, grey slurry coating became a brittle, light brown covering

Initial mass: 2.45133 g
 Final mass: 2.44835 g
 Mass of spall: 0 g
 Total mass: 2.44835 g

Sample OX10 Coated with Slurry (Y/N) Mass per unit area .274 mg/mm²

Furnace Setpoint: 1050 °C
 Furnace Temperature: 1051 °C
 Duration of exposure: 4 hrs.
 Start time-date: 9:45 - 6/6/07
 End time-date: 1:45 - 6/6/07

Details/Results:

The powdery, grey slurry coating became a brittle, light brown covering

Initial mass: 2.51517 g
 Final mass: 2.50770 g
 Mass of spall: .00225 g
 Total mass: 2.50985 g

K. J. Chiang 6/18/07

12 June 2007 M.P.

Sample OX9 Coated with Slurry (Y/N). Mass per unit area .215 mg/mm²

Furnace Setpoint: 1150 °C
 Furnace Temperature: 1149 °C
 Duration of exposure: 2 hrs.
 Start time-date: 8:30 - 6/7/07
 End time-date: 10:30 - 6/7/07

Details/Results:
The slurry coating began to drip on one side of the sample. The slurry turned to a shiny black color, the exposed oxidized metal became black (dull).

Initial mass: 2.44835 g
 Final mass: 2.40903 g
 Mass of spall: .00426 g
 Total mass: 2.41329 g

Sample OX11 Coated with Slurry (Y/N). Mass per unit area .238 mg/mm²

Furnace Setpoint: 1200 °C
 Furnace Temperature: 1200 °C
 Duration of exposure: 2 hrs.
 Start time-date: 10:40 - 6/7/07
 End time-date: 12:40 - 6/7/07

Details/Results:
The oxidized metal was dull black and the dried slurry was shiny black. Some spall could not be removed from the bottom of the crucible.

Initial mass: 2.43478 g
 Final mass: 2.41167 g
 Mass of spall: .00255 g
 Total mass: 2.41522 g

Sample OX12 Coated with Slurry (Y/N). Mass per unit area .267 mg/mm²

Furnace Setpoint: 1200 °C
 Furnace Temperature: 1200 °C
 Duration of exposure: 20 hrs.
 Start time-date: 3:15 - 6/7/07
 End time-date: 11:15 - 6/8/07

Details/Results:
Structural integrity was not maintained. Only a small portion of the sample remained in fact. There was some discoloration inside the crucible.

Initial mass: 2.46458 g
 Final mass: 1.07665 g
 Mass of spall: 1.74477 g
 Total mass: 2.82142 g

K. J. Ching 6/18/07

Sample OX13 Coated with Slurry (Y/N). Mass per unit area _____

Furnace Setpoint: 1200 °C
 Furnace Temperature: 1200 °C
 Duration of exposure: 20 hrs.
 Start time-date: 3:15 - 6/7/07
 End time-date: 11:15 - 6/8/07

Details/Results:
The sample was oxidized, thus, its color changed to dull black. There was some discoloration on the inside of the crucible.

Initial mass: 2.26255 g
 Final mass: 2.16415 g
 Mass of spall: .09212 g
 Total mass: 2.25627 g

Sample OX14 Coated with Slurry (Y/N). Mass per unit area _____

Furnace Setpoint: 1200 °C
 Furnace Temperature: 1200 °C
 Duration of exposure: 4 hrs.
 Start time-date: 9:20 - 6/8/07
 End time-date: 1:20 - 6/8/07

Details/Results:
The sample was oxidized, thus, its color changed to dull black. There was some discoloration in parts of the crucible.

Initial mass: 2.34129 g
 Final mass: 2.34392 g
 Mass of spall: .00056 g
 Total mass: 2.34448 g

12 June 2007 M.P.

K. J. Ching 6/18/07

15 June 2007 M.P.

Summary of Mass Changes Throughout C-22 Coupon Experiments

Sample #	OX9	OX10	OX11	OX12	OX13	OX14
Height (mm)	14.18	14.19	14.31	14.27	14.23	14.48
Width (mm)	12.58	12.64	12.56	12.33	12.45	12.46
Thickness (mm)	1.56	1.58	1.53	1.57	1.51	1.53
Initial Wt. (g)	2.35501	2.39145	2.32792	2.34628	2.26255	2.34129
Wt. after 20h at 900C	2.35594	2.39244	2.32883	2.34735	N/A	N/A
Difference in wt. after 20h 900C	0.00093	0.00099	0.00091	0.00107	N/A	N/A
Wt. after coating with slurry	2.45133	2.51517	2.43478	2.46458	N/A	N/A
Difference in wt. after coating	0.09539	0.12273	0.10595	0.11723	N/A	N/A
Area (mm ²)	444.0802	447.4267	445.3588	439.2926	438.4694	446.9474
Wt. Change per unit area (g/mm ²)	0.000215	0.000274	0.000238	0.000267	N/A	N/A
Wt. before 2h at 1050C (g)	2.45133	N/A	2.43478	2.46458	N/A	N/A
Wt. after 2h at 1050C (g)	2.44835	N/A	N/A	N/A	N/A	N/A
Difference in wt. after 2h at 1050C	-0.00298	N/A	N/A	N/A	N/A	N/A
Wt. before 4h at 1050C (g)	N/A	2.51517	N/A	N/A	N/A	N/A
Wt. after 4h at 1050C (g)	N/A	2.50995	N/A	N/A	N/A	N/A
Difference in wt. after 4h at 1050C	N/A	-0.00522	N/A	N/A	N/A	N/A
Wt. before 2h at 1150C (g)	2.44835	N/A	N/A	N/A	N/A	N/A
Wt. after 2h at 1150C (g)	2.41329	N/A	N/A	N/A	N/A	N/A
Difference in wt. after 2h at 1150C	-0.03506	N/A	N/A	N/A	N/A	N/A
Wt. before 2h at 1200C (g)	N/A	N/A	2.43478	N/A	N/A	N/A
Wt. after 2h at 1200C (g)	N/A	N/A	2.41522	N/A	N/A	N/A
Difference in wt. after 2h at 1200C	N/A	N/A	-0.01956	N/A	N/A	N/A
Wt. before 20h at 1200C (g)	N/A	N/A	N/A	2.46458	2.26255	N/A
Wt. after 20h at 1200C (g)	N/A	N/A	N/A	2.82142	2.25627	N/A
Difference in wt. after 20h at 1200C	N/A	N/A	N/A	0.35684	-0.00628	N/A
Wt. before 4h at 1200C (g)	N/A	N/A	N/A	N/A	N/A	2.34129
Wt. after 4h at 1200C (g)	N/A	N/A	N/A	N/A	N/A	2.34448
Difference in wt. after 4h at 1200C	N/A	N/A	N/A	N/A	N/A	0.00319

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OX9

500x



1000x



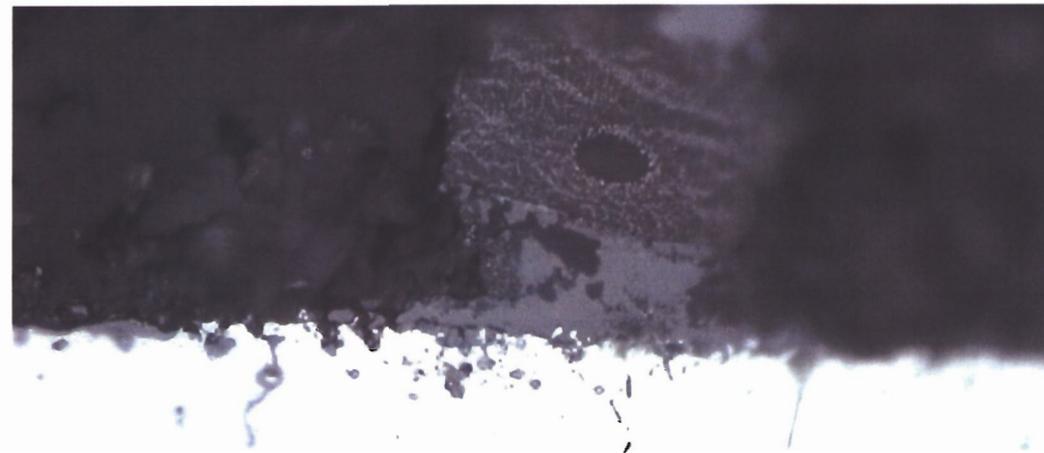
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19 June 2007 M.P.

OX 10

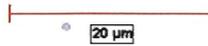


500x



1000x

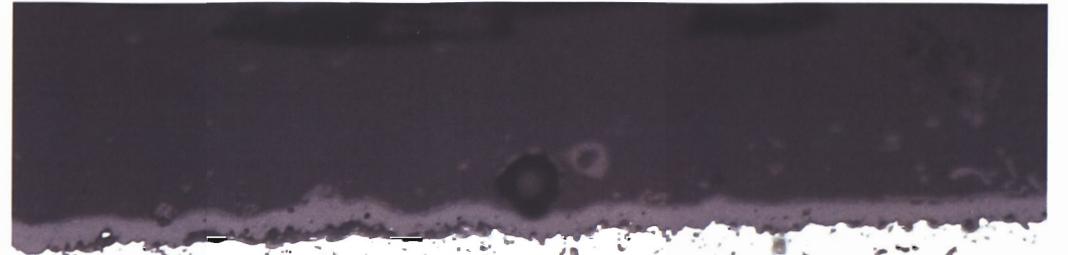
OX-10



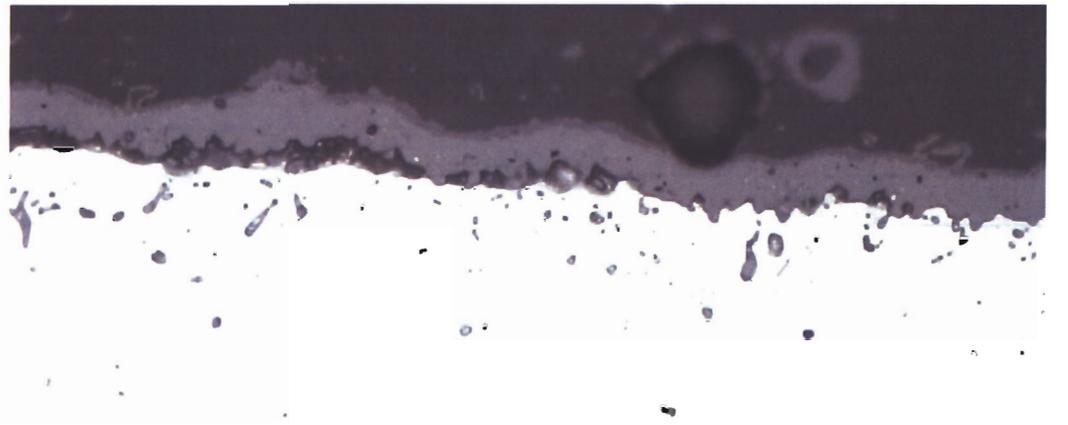
K. T. Chuang 6/25/07

19 June 2007 M.P.

OX 11



500x



1000x

OX-11



K. T. Chuang 6/25/07

19 June 2007 M.P.

OX 12

500x



1000x



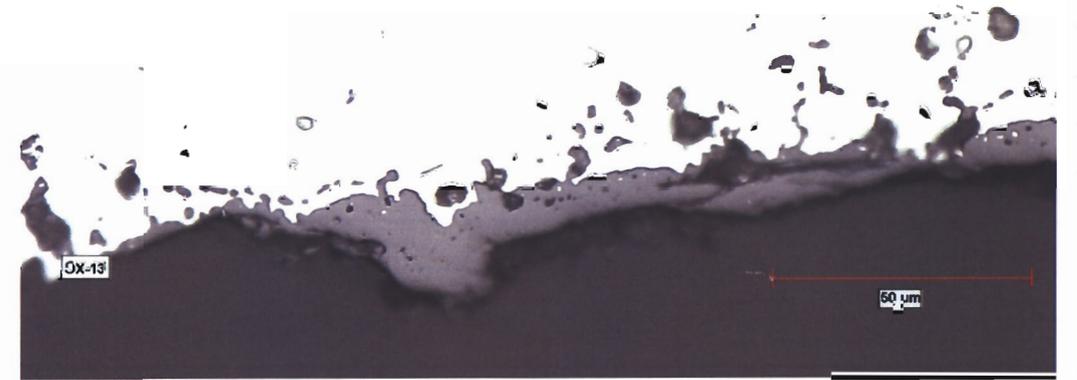
K. J. Chirif

6/25/07

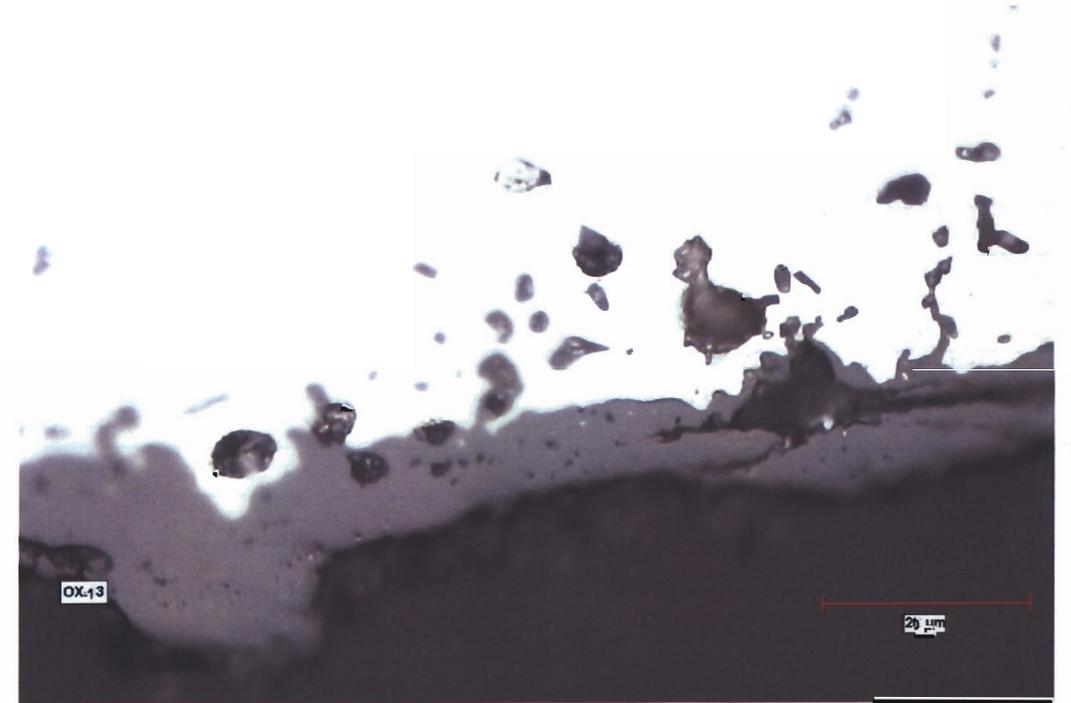
19 June 2007 M.P.

OX 13

500x



1000x

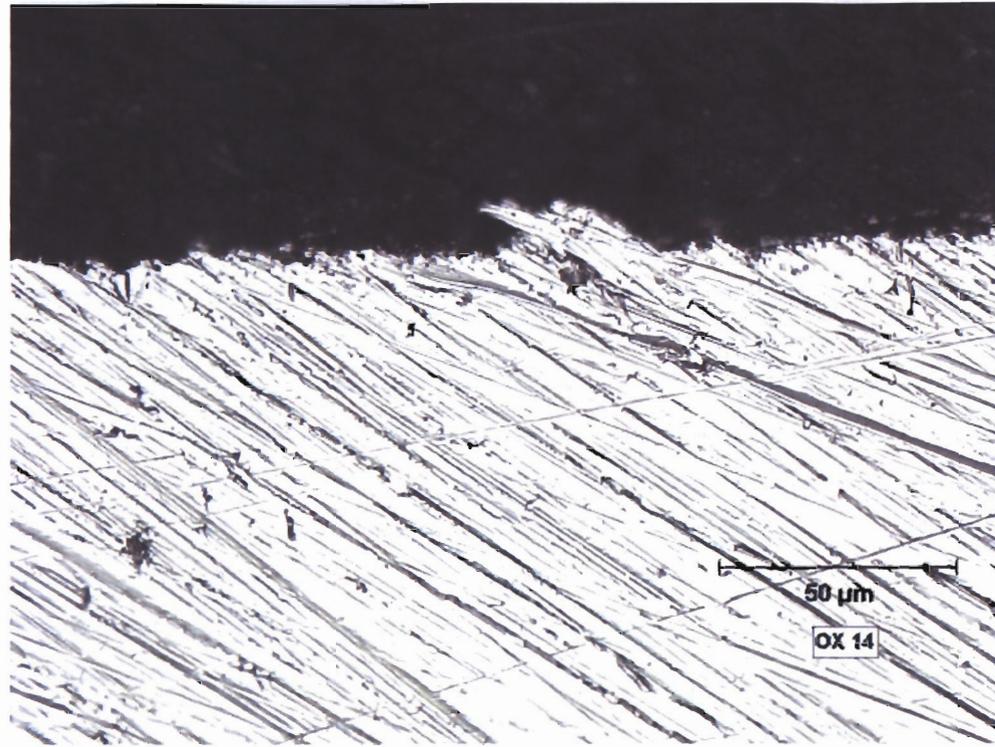


K. J. Chirif

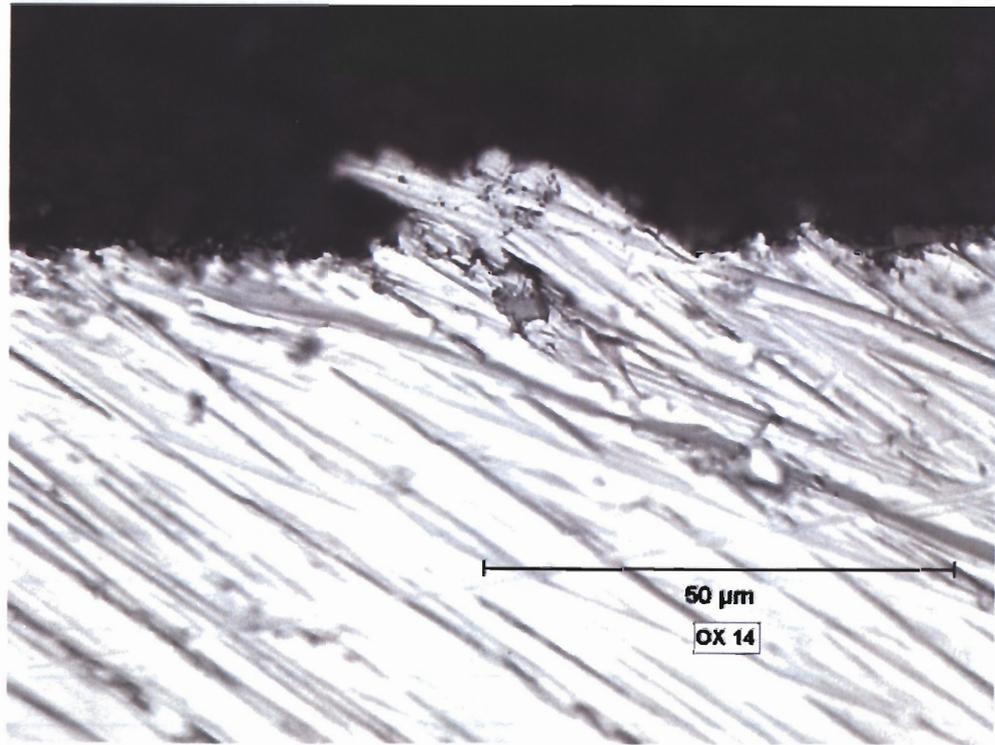
6/25/07

19 June 2007 M.P.

OX 14



500x



1000x

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6/25/07

20 June 2007 M.P.

Oxygen Attack and Scale Measurements

Sample	Maximum Oxygen Penetration (μm)	External Scale Depth (μm)
OX9	11.82	15.51
OX10	10.93	4.18
OX11	13.29	6.56
OX12	28.08	7.15
OX13	28.96	7.75
OX14	N/A	N/A

*OX 14 was too scratched to accurately measure these values
-several attempts were made to re-polish the specimen
but the scratches remained.

Starrett Digital Calipers used to make image measurements

20 June 2007 M.P.

K.T. Ching

6/25/07

21 June 2007 M.P.

Lava Rock Melting Tests

Task Assigned to: Matthew J. Panariello

Objective: To test the ability of Alloy C-22 (in crucible form) to withstand exposure to magma from different volcanic sites, and to analyze the extent of structural failure, if any.

Equipment/Materials:

- Hastelloy C-22 crucibles
HT#: 2277-5-3299
Major element composition: 58% Ni, 22% Cr, 13% Mo, 4% Fe, 3% W
Machined dimensions (approx.): length= 1.5 in, diameter= 1 in, bore= 1 in
- 200 mL beaker (2)
- Acetone
- Hawaii lava rock samples (HI #1 & HI #2)
- Lathrop Wells lava rock sample
- Distilled Water
- Fisher twist-tie sample bags
- Steel tongs
- Tweezers
- Heat resistant safety gloves
- Safety glasses
- Fisher Scientific FS 14 Ultrasonic Cleaner
- Cold epoxy mounting kit
- 60, 120, 240, 320, 600 grit adhesive backed 8" diameter abrasive discs
- Buehler Ecomet 3 variable speed grinder/polisher
120 volt
490 Max RPM
Cat #: 60-1960-160
sn: 462-A3P-0208
- Buehler Isomet Plus precision wet cut-off saw
115 volt
4500 Max RPM
Cat #: 11280-160
sn: 452-I5P-835
- CM Inc. Rapid Temp. Furnace
Max temp: 1700°C
sn: 029786
Model #: 98002-24
- Lindberg Oven
Max temp: 1200°C
sn: 909172
Model #: 51333
- Sartorius Genius Scale (Analytical Balance)
Mass range: .00001g to 200g
sn: 12809099
Calibration due: Nov 11, 07

Identified Sources of Error:

- Mass lost from spalling during heating and cooling
- Any difference in chemical or physical composition of different samples
- Any impurities that may have been encountered by the samples between heating and storing

K. J. Ching
6/25/07

21 June 2007 M.P.

Procedure:

1. Preparation of Alloy C-22 Crucible and Lava Rock:

Before the experiments are run, the crucibles of C-22 are weighed using the analytical balance. They are then placed into a 200 mL beaker with acetone and placed in the ultrasonic cleaner for approximately 6 minute. The lava rock sample is broken into fragments (approx. pencil-eraser sized) and used to fill the cleaned crucible. Finally, the crucible and lava rock are weighed together to attain the mass of lava rock added to the crucible.

2. Heating HI #1 Lava Rock to Melting Temps:

The specimen is placed into the furnace at a predetermined starting point from which the temperature is increased by intervals of 20°C every ~1 hour. Once the melting temperature is reached and the lava rock is in its molton state, the specimens will remain at these conditions for a set period of time (to be determined during test). Upon removal, the specimen is allowed to cool to 600°C in the Lindberg Oven for 30 min. and then to room temperature for another 30 min.

3. Heating HI #2 and LW 4-4 Lava Rocks Comparitively:

Both specimens are to remain under identical conditions once the experiment is started. They are placed side by side in the furnace at a set starting temperature and gradually heated as in the HI #1 heating experiment. Again, upon both specimens reaching the melting point and becoming molton, they are left under those conditions for a set period of time (to be determined). When heating is completed, the specimens are gradually cooled as in the HI #1 heating experiment.

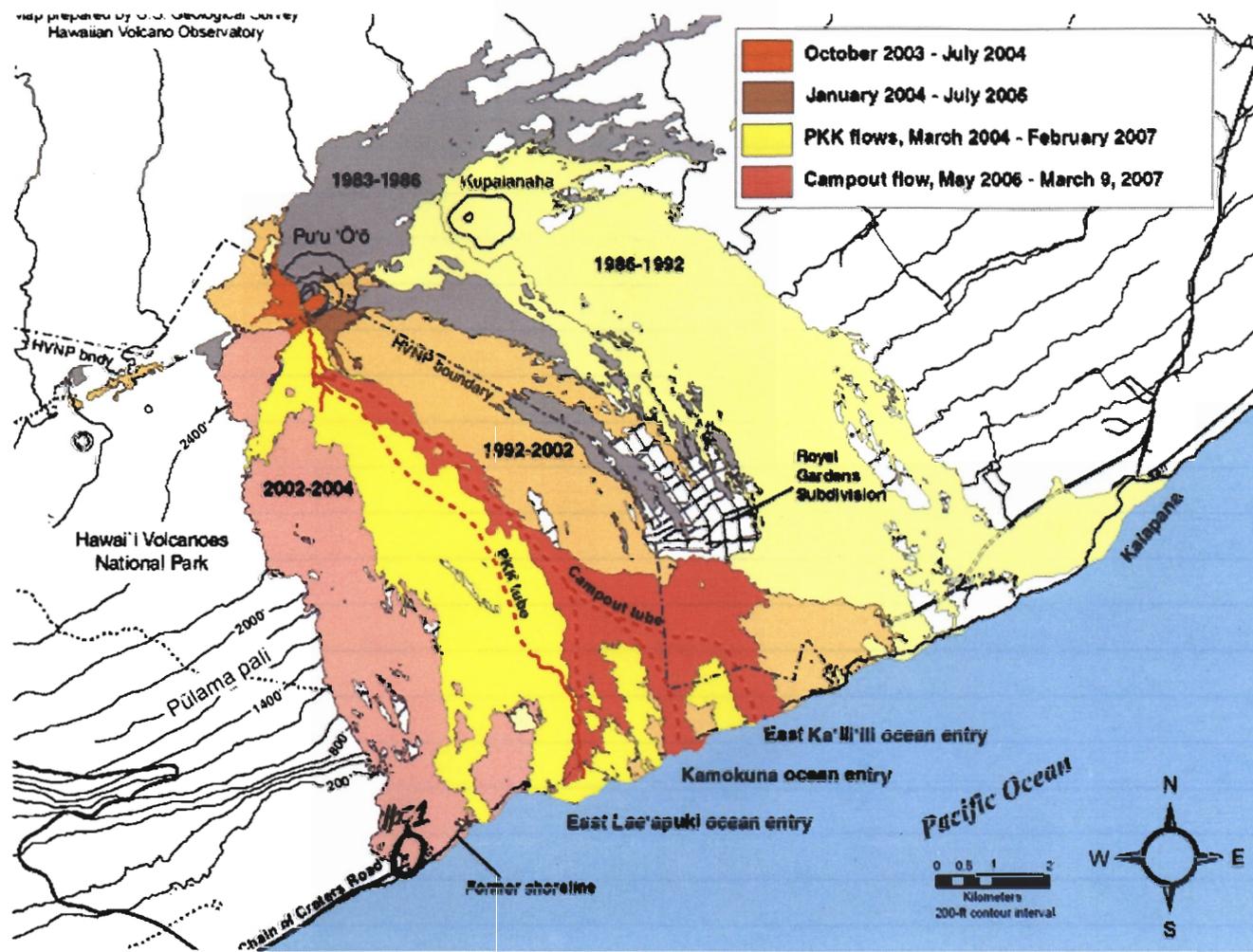
4. Data Analysis:

The crucibles, once cooled and reweighed, are cut into quarter sections so that the interactions between the C-22 and magma are exposed. These pieces are photographed under a low magnification (.6 or .8), and then set in epoxy mounts and polished following the procedure described on pages 20 and 21 of this notebook. They are then photographed under higher magnification (50x to 500x). Some specimens may be analyzed under a Scanning Electron Microscope to learn the material composition and etched to determine grain structure.

21 June 2007 M.P.

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Lava Rock Sample HI #1

Location: Hawaii

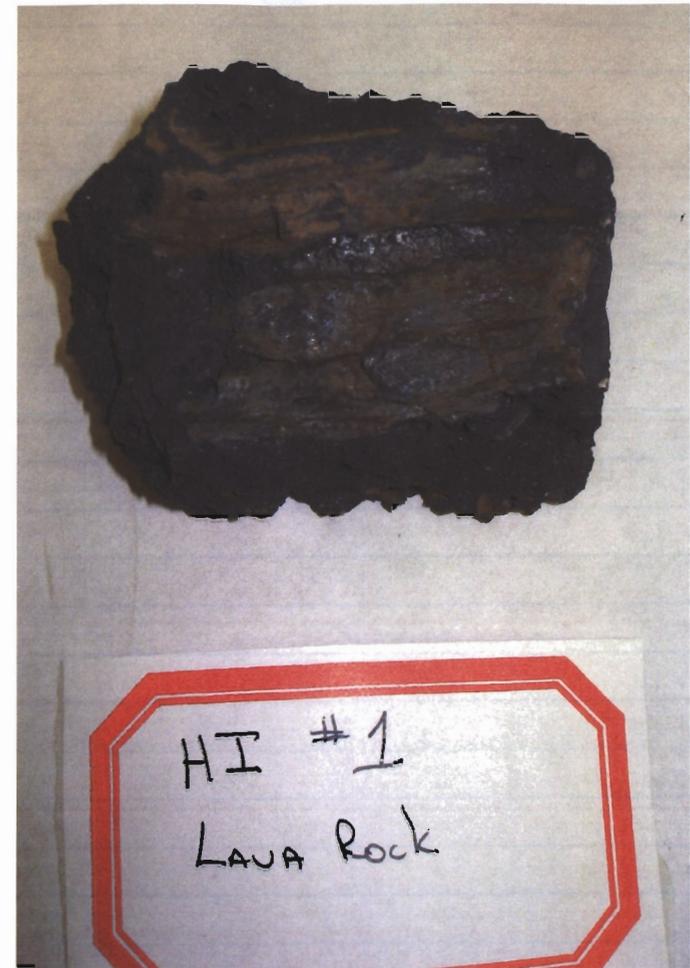
Coordinates (Based on NAD 83 GPS system):
0180419
213 5277

Taken from a 2002-2004 flow.

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28 June 2007 M.P.



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Yucca Mountain Rock Testing

OVEN= CM Inc. Rapid Temp. Furnace
SN: 48002-24
OVEN SETPOINT= 1050°C
OVEN TEMPERATURE= 1042.5°C

Specimen: C-22 Crucible HT# 2277-5-3294 st. wt. = 106.7103g
Rock HZ #2

start weight: 116.7317g weight of Rock = 10.0214g
end weight: Large piece = 84.92227g Small piece = 14.53513g Spall = 23.88964

measurement scale: Sartorius Genius Scale SN#: 1250099 Cal: 5/11/07 Due: 11/11/07

Measurement taken with OMEGA MICROPROCESSOR THERMOMETER MODEL# HH22

Thermocouple= SN#= 328 CAL= 4/26/07 DUE= 10/26/07
SN#= 328 CAL= 4/5/07 DUE= 8/5/07

AMOUNT OF TIME = Start time: 6/14/07 @ 3:30 pm
End time: 6/18/07 @ 8:30 am

DETAILS=

6/15/07 @ 8:00 am: setpoint = 1030°
Temp. = 1078.5°

No change

@ 9:00 am: setpoint = 1100°
Temp. = 1095.5°

specimen glowing red

@ 10:00 am: setpoint = 1120°
Temp. = 1112.2°

No change

@ 11:00 am: setpoint = 1160°
Temp. = 1147.5°

Rocks at the edge beginning to fuse

@ 12:00 pm: setpoint = 1180°
Temp. = 1166.5°

Rock is molten but still solid

@ 1:00 pm: put into Lindberg oven @ 600°C
Rock is completely molten (magma)

@ 1:30 pm: oven temp is dropped to 300°C

@ 1:50 pm: - procedure change
specimen is put back into Furnace @ 1164°C

@ 3:00 pm: Decision made to leave specimen in furnace over the weekend.

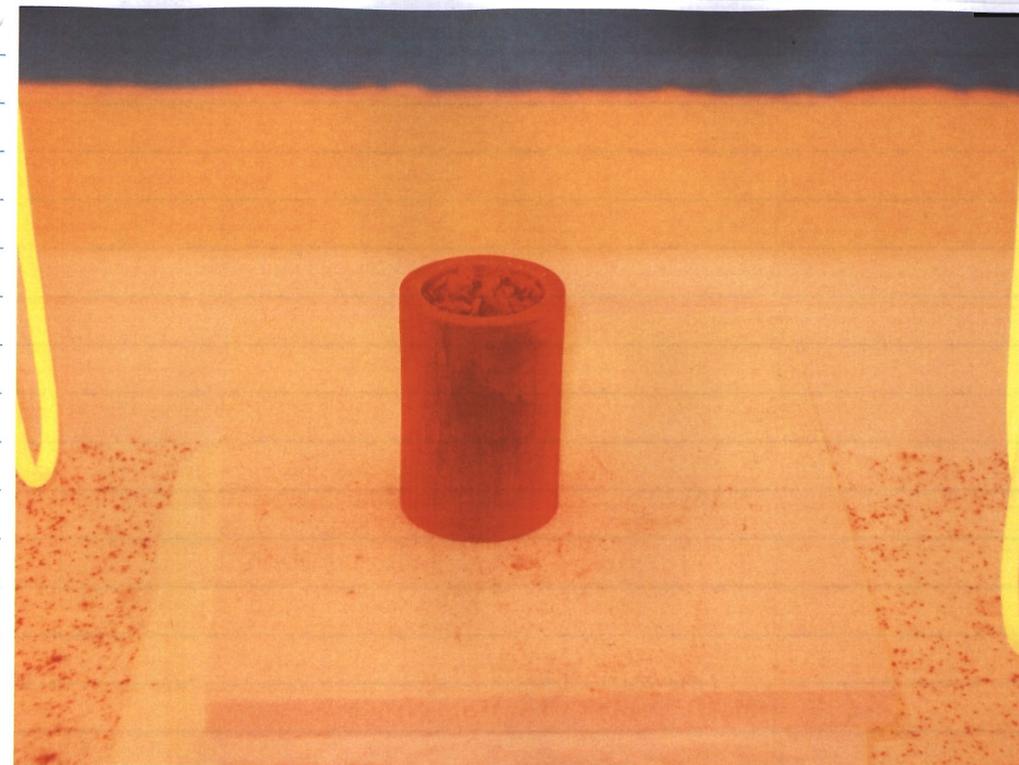
6/18/07 @ 8:30 am: specimen is put into Lindberg oven @ 600°C

@ 9:50 am: specimen is pulled out of the oven to cool to room temp.

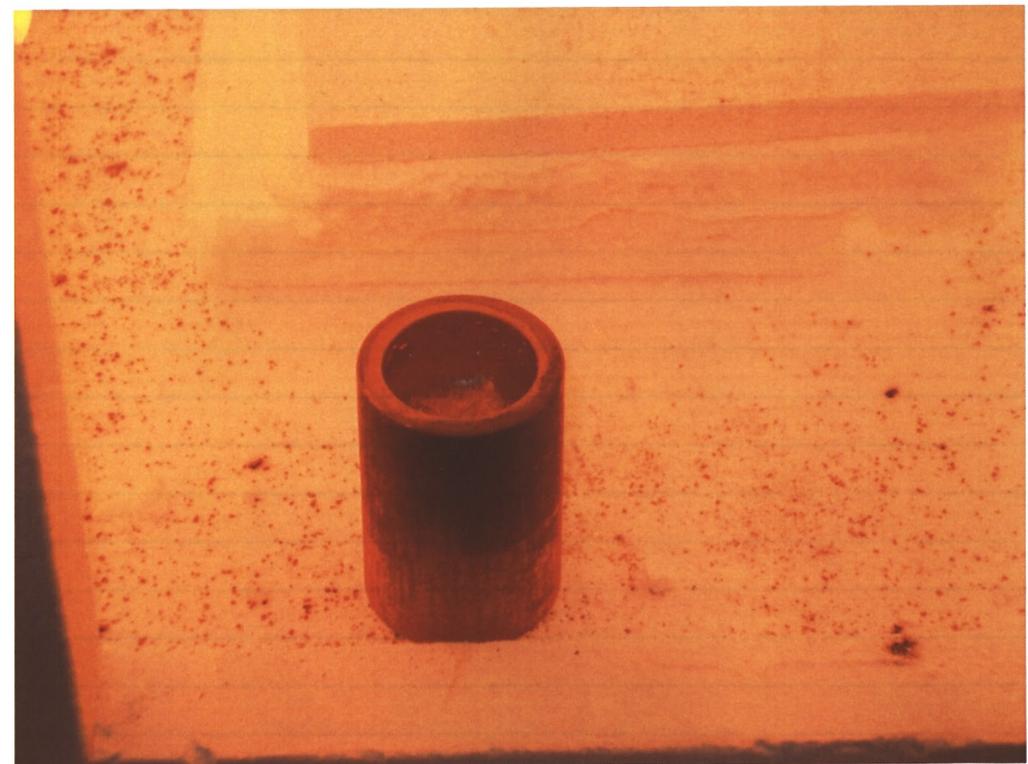
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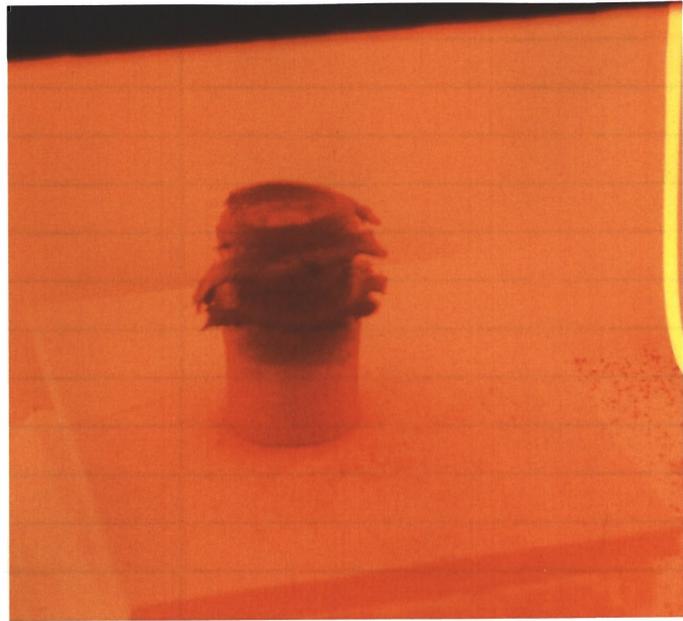


6/15/07 - Picture taken prior to 9:00 checkpoint (see previous page)
Lava Rock still in solid state



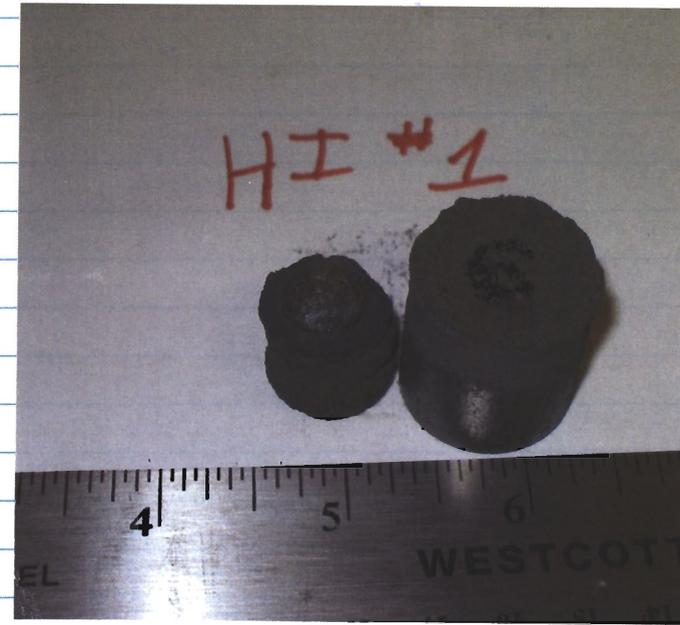
6/15/07 - Picture taken prior to 3:00 checkpoint (see previous page)
K.J. Chirif 7/6/07 Molten state achieved.

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6/18/07 - Picture taken just before crucible removed from furnace.

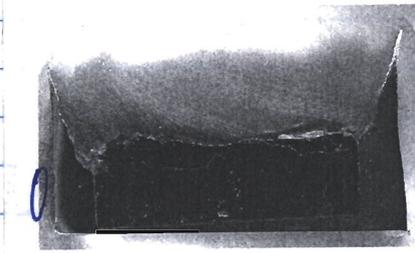
Picture taken while crucible was cooling to room temperature. (Spalling has begun)



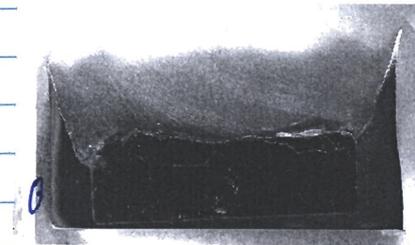
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Pictures taken after spalling was complete and top half of crucible had fallen off.

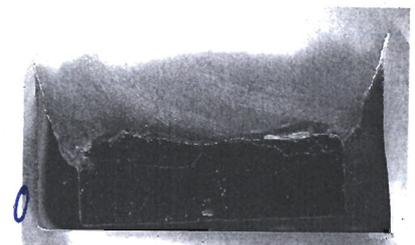
29 June 2007 M.P. PS



50x
Remaining C-22
Crucible + Solidified
HI #1 Magma



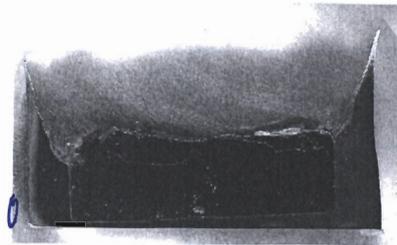
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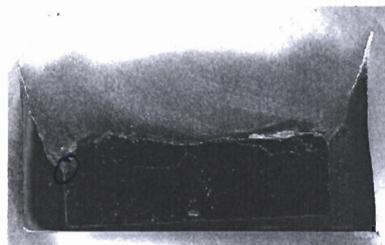
100x
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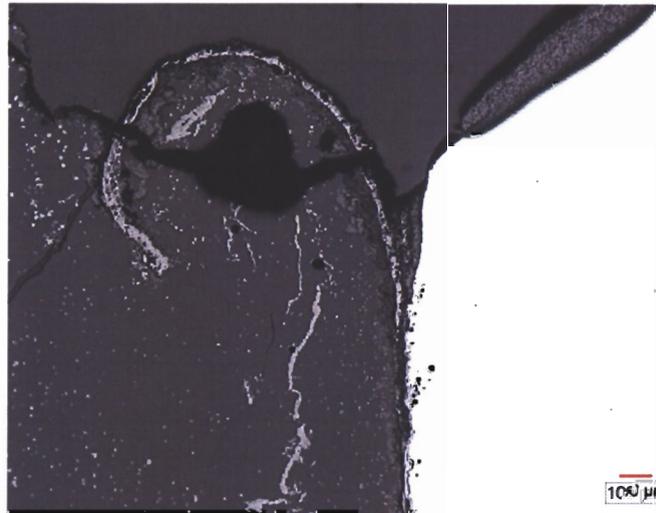
29 June 2007 M.P.



200X



50x

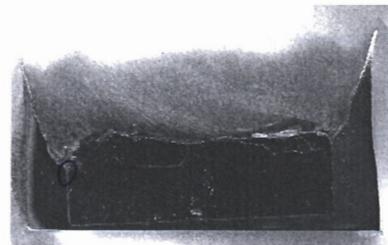


100X

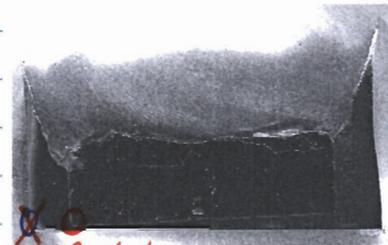


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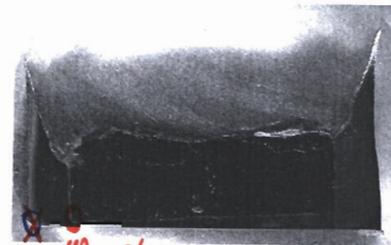
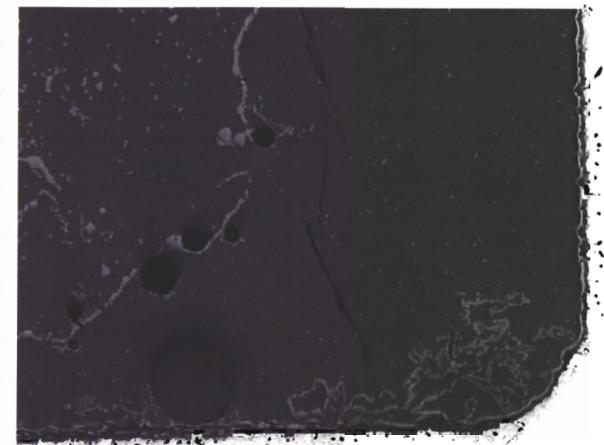
29 June 2007 M.P.



200X



50x

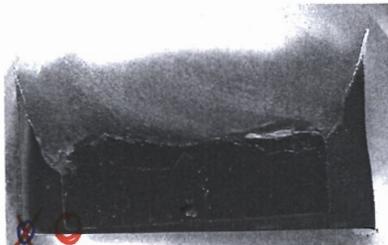


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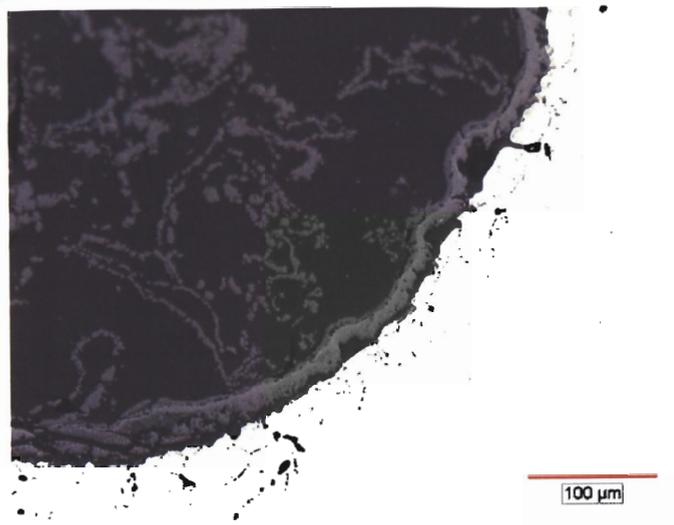
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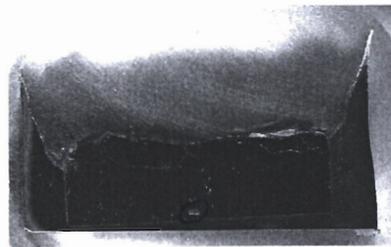


M.P. 7/20/07

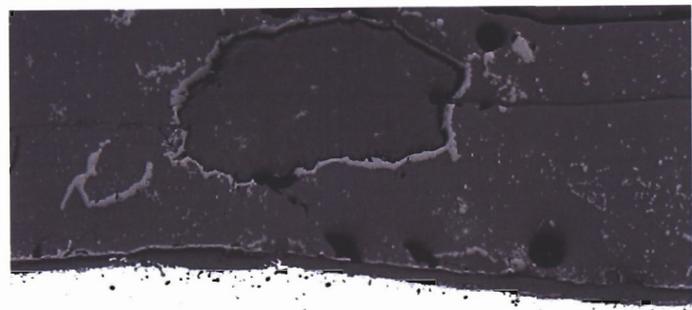
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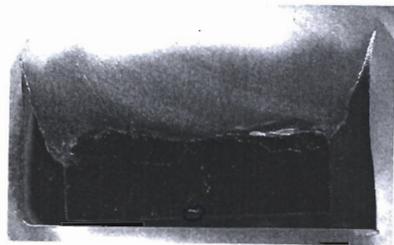
100 µm



50x

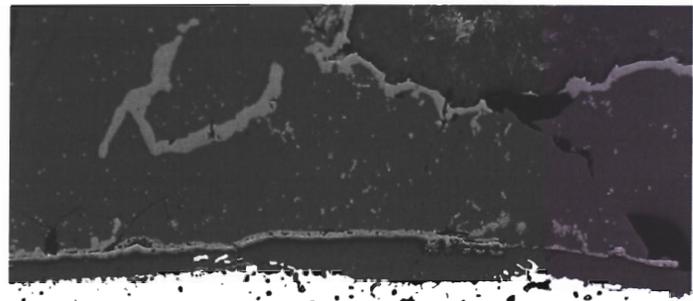


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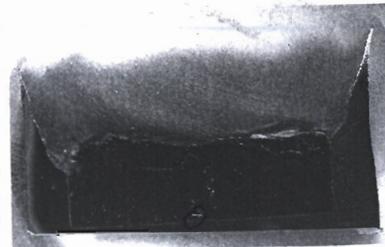
100x

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100 µm

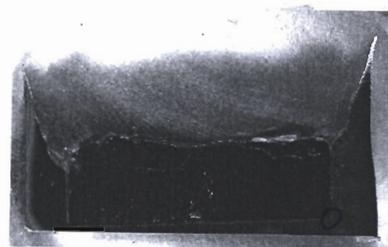
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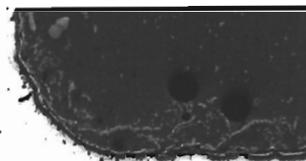
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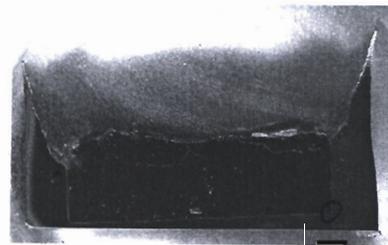
100 µm



50x

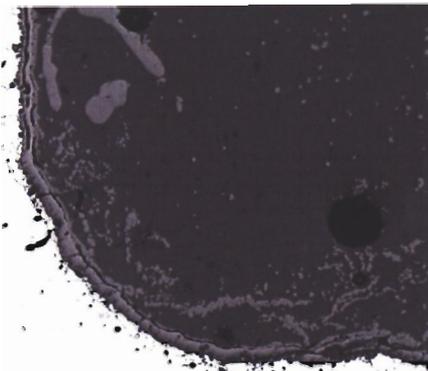


100 µm



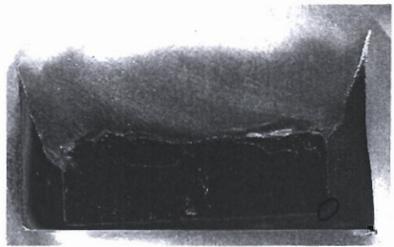
100x

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7/6/07



100 µm

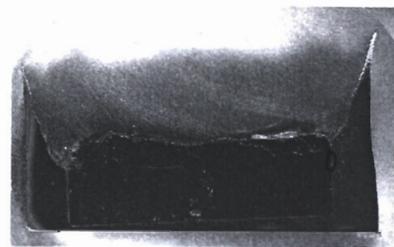
29 June 2007 M.P.



200x



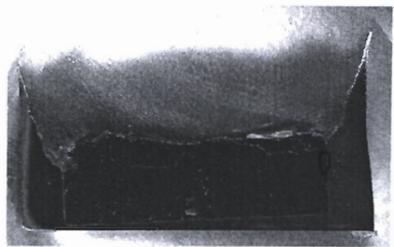
100 μm



50x

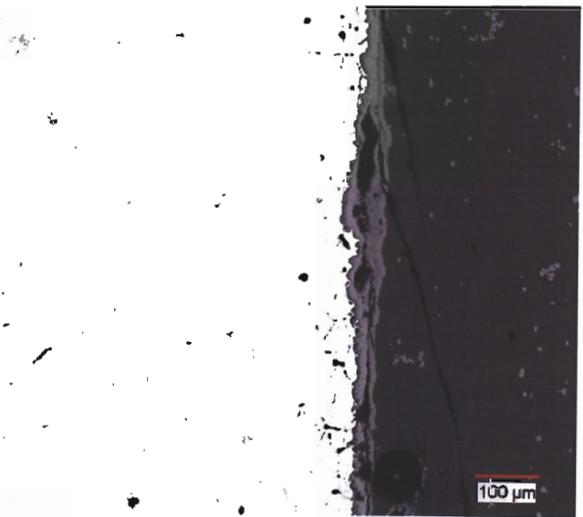


100 μm



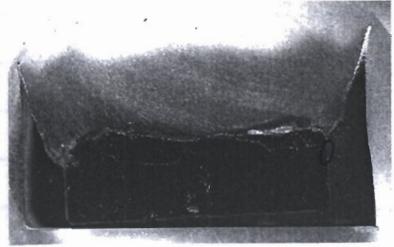
100x

K-J. Chief
7/6/07



100 μm

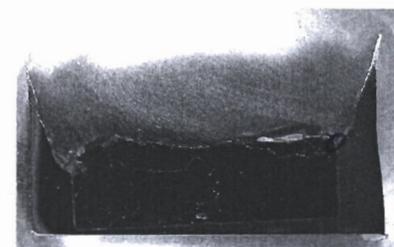
29 June 2007 M.P.



200x



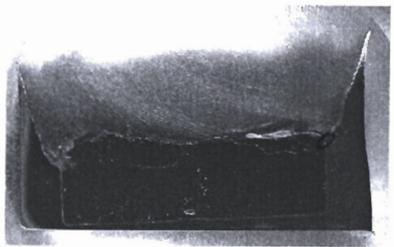
100 μm



50x



100 μm



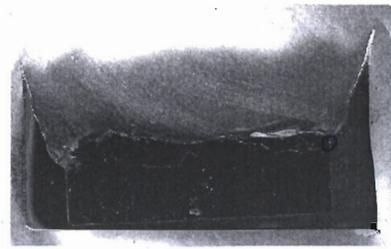
100x

K-J. Chief
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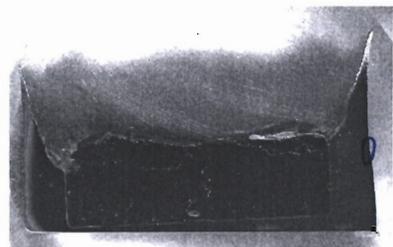


100 μm

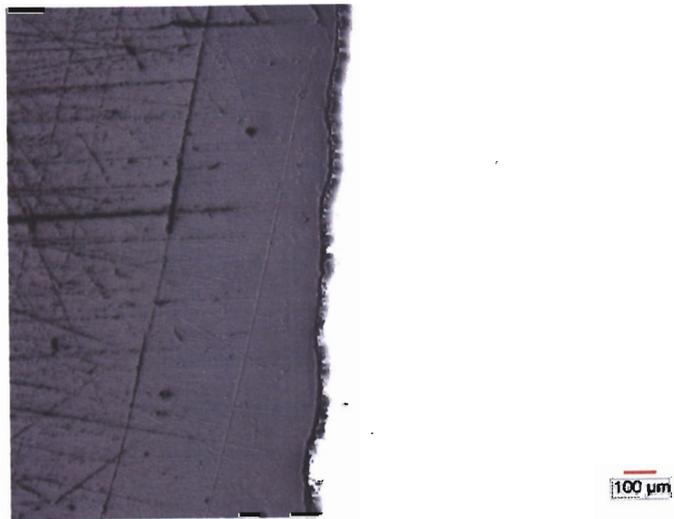
29 June 2007 M.P.



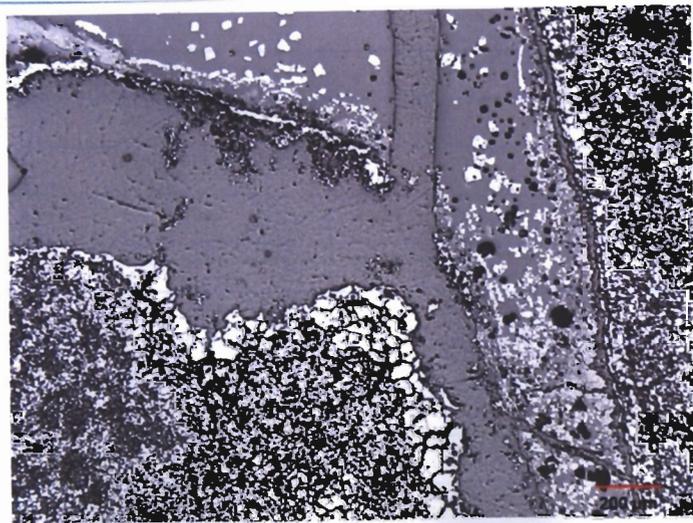
200x



50x

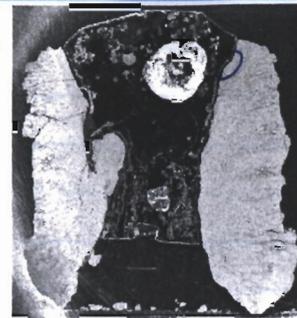


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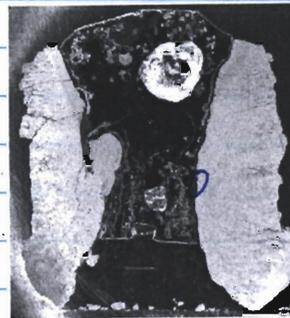
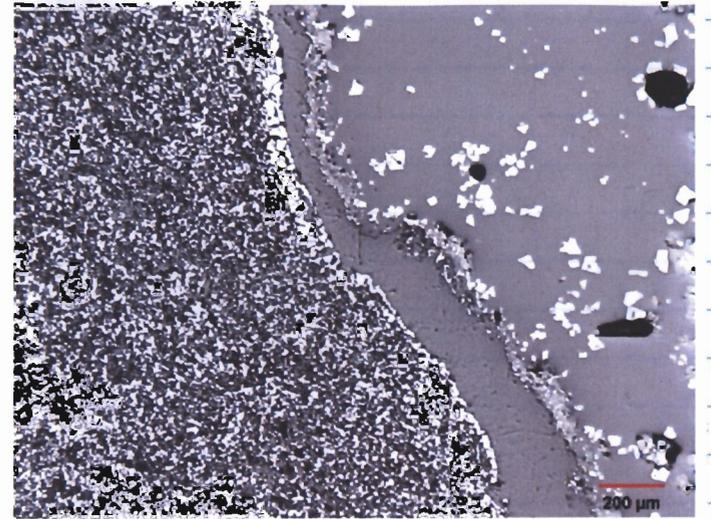


K. J. Chouf
7/6/07
Solidified HI#1 Magna
C-22 + HI#1 Reaction
Product

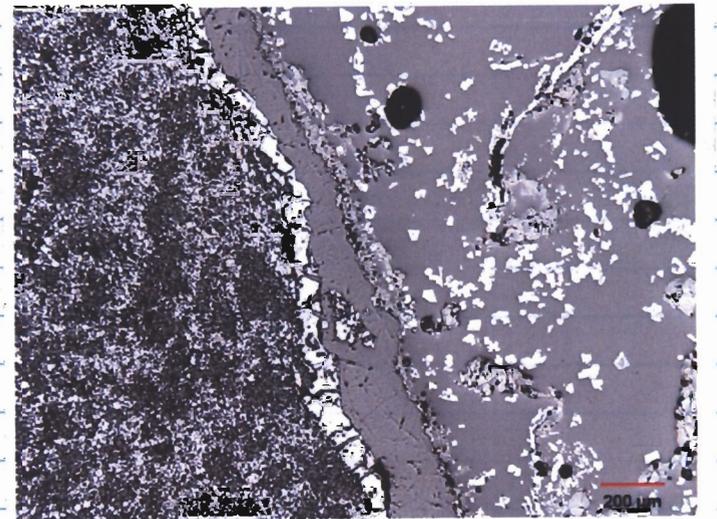
29 June 2007 M.P.



50x



50x

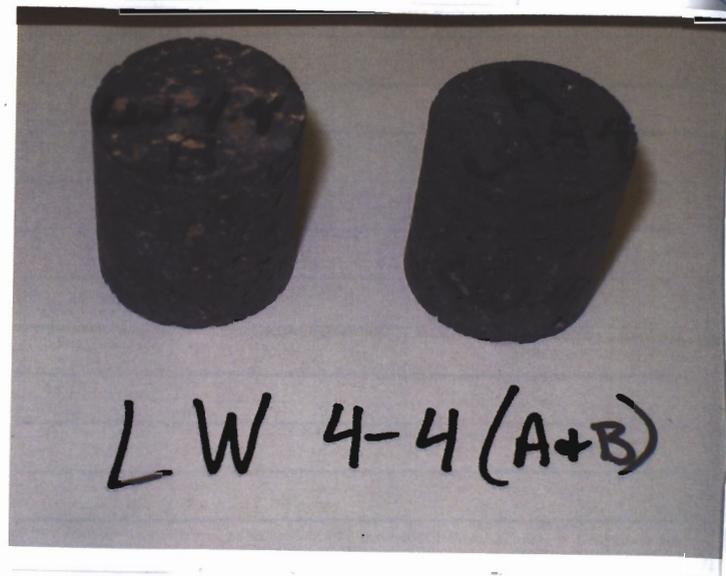
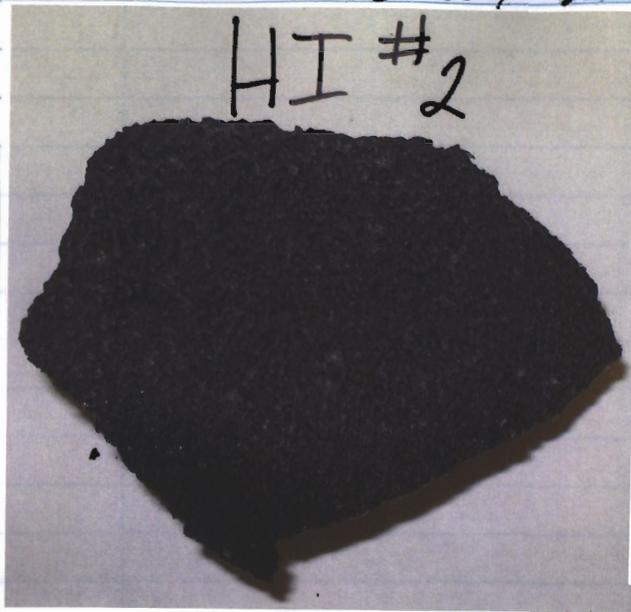


~~29 June 2007 M.P.~~

~~K. J. Chouf~~

~~7/6/07~~

5 July 2007 M.P.



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7/6/07

5 July 2007 M.P.

Yucca Mountain Rock Testing

OVEN=
CH Inc. Rapid Temp Furnace
SN: 99002-24

OVEN SETPOINT= 900°C
OVEN TEMPERATURE= 901.5°C

Specimen: C-22 crucibles HI# 2277-5-2299 st. wt. = 103.3278g (LW4-4), 105.7592g (HI#2)
Rock LW4-4, Rock HI#2

start weight: LW4-4: 113.34243g HI#2: 113.92514g
end weight: LW4-4: Top= 9.03737g, Bottom= 93.13353g, Spill= 19.37502g HI#2: Crucible= 107.35120g, Spill= 7.56536g

measurement scale: Sartorius Genius scale SN#: 12809099 Cal: 5/11/07 Due: 11/11/07

Measurement taken with OMEGA MICROPROCESSOR THERMOMETER MODEL# HH22
SN# = T-94140 CAL = 4/26/07 DUE = 10/26/07
Thermocouple = SN# = 328 CAL = 1/5/07 DUE = 8/5/07

AMOUNT OF TIME = Start time: 6/20/07 @ 10:00 am
End time: 6/21/07 @ 2:30 pm

DETAILS=

6/20/07 @ 10:00 am - specimen were put into the furnace and set to 1100°C and left to stabilize at 1090.2°C

@ 11:00 am - 1120°C = setpoint
Temp = 1102.9°C
2 pictures taken - HI#2 Rock appears to be fusing

@ 12:00 pm - setpoint = 1140°C
Temp = 1127.4°C
2 pictures taken

@ 1:30 pm - setpoint = 1160°C
Temp = 1147.2°C
2 pictures taken - LW4-4 is in molten state

@ 2:30 pm - setpoint = 1180°C
Temp = 1165.3°C
2 pictures taken - LW4-4 has completely melted
HI#2 has reached molten state

* Thermocouple discovered to be giving an inaccurate reading on last check point (6/21/07 @ 8:30 am) when checked against another thermocouple. Replacement thermocouple: SN# = 12353 Cal = 7/20/06 Due = 7/16/07

6/21/07 @ 8:30 am - Temp = 1188.8°C
(using new thermocouple)

2 pictures taken - significant scale has formed on LW4-4 crucible

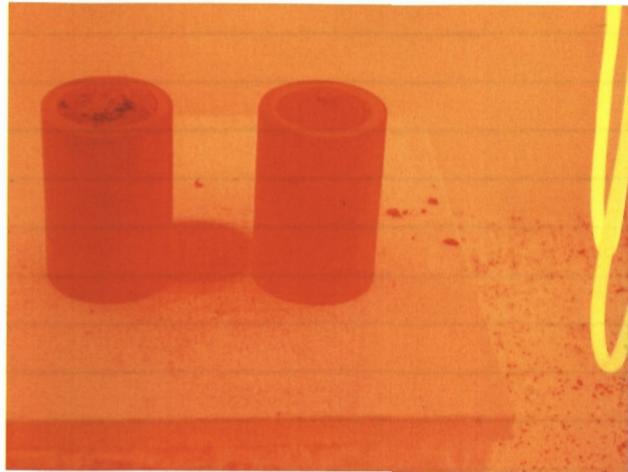
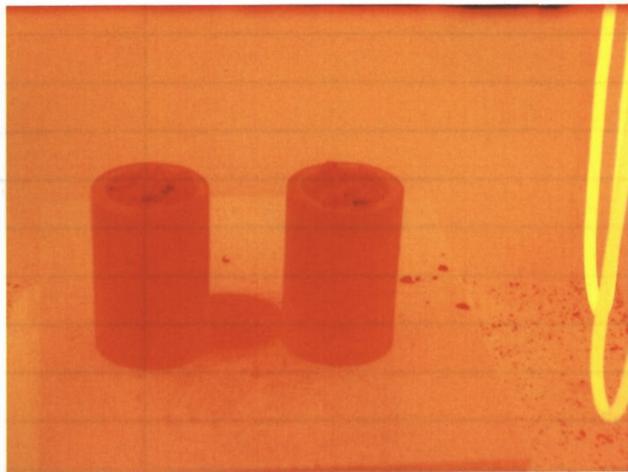
@ 11:30 am - Temp = 1186.4°C
2 pictures taken - Heavily oxidized layers of C-22 Metal appear to be peeling back from the top of the LW4-4 crucible.

@ 2:30 pm - Temp = 1188.6°C
1 picture taken - specimens were placed into the Lindberg oven @ 600°C for gradual cooling

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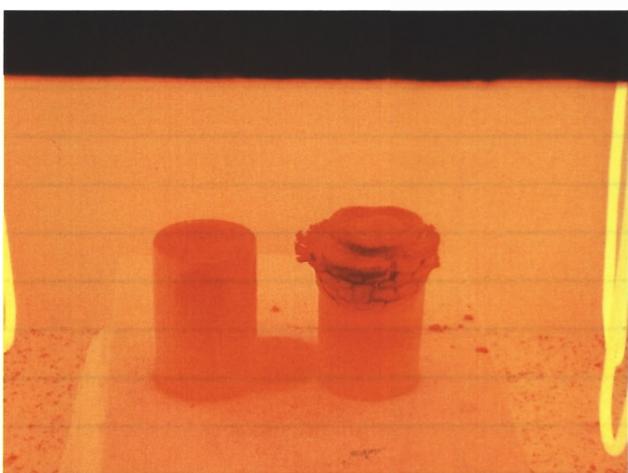
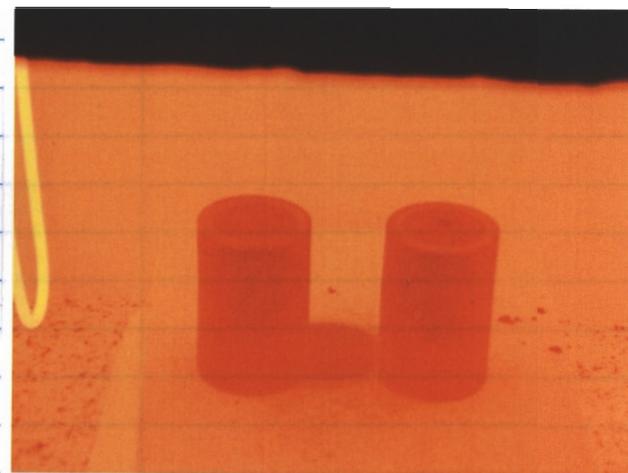
7/6/07

9 July 2007 M.P.



Picture taken on 6/20 prior to the 12:00 checkpoint

Picture taken on 6/20 prior to the 1:30 checkpoint - LW4-4 had reached molten state.



Picture taken on 6/20 prior to the 2:30 checkpoint - HI#2 had reached molten state

Picture taken on 6/21 prior to removing crucibles from the furnace at 2:30

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Picture taken while crucibles were cooling to room temperature

Top view while cooling - LW4-4 had begun spalling



LW4-4 had finished spalling - the top half of the crucible had fallen off

HI#2 with spall from inside the crucible

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12 July 2007 M.P.



HI #2 Crucible



LW 4-4 Crucible

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Original Wall Thickness = 3.20 mm

Height From Base (mm)	HI #2 Wall Thickness (mm)	LW 4-4 Wall Thickness (mm)
0	3.20	3.17
2	2.94	2.97
4	2.12	2.94
6	1.68	2.91
8	1.27	2.87
10	1.34	2.52
12	1.38	1.97
14	1.92	0.77
16	2.18	0.00
18	2.34	0.00
20	2.40	0.00
22	2.55	0.00
24	2.81	0.00

Height From Base (mm)	HI #2 Wall Thickness (mm)	LW 4-4 Wall Thickness (mm)
0	3.20	3.20
2	2.79	3.12
4	2.62	3.08
6	2.22	3.10
8	1.85	3.02
10	1.50	2.55
12	1.66	2.12
14	1.92	1.09
16	2.00	0.54
18	2.48	0.00
20	2.79	0.00
22	3.10	0.00
24	3.08	0.00

Left Wall

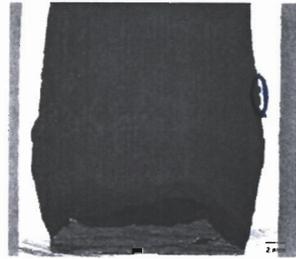
Right Wall

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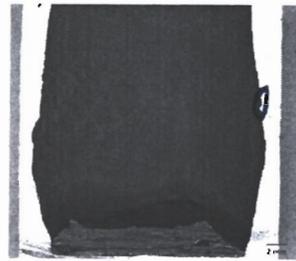
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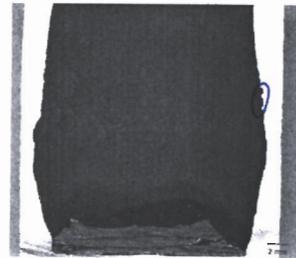
16 July 2007 M.P.
HI #2 Crucible
Temperature: 1180°C
Environment: Air
Time: 24hr.



50x

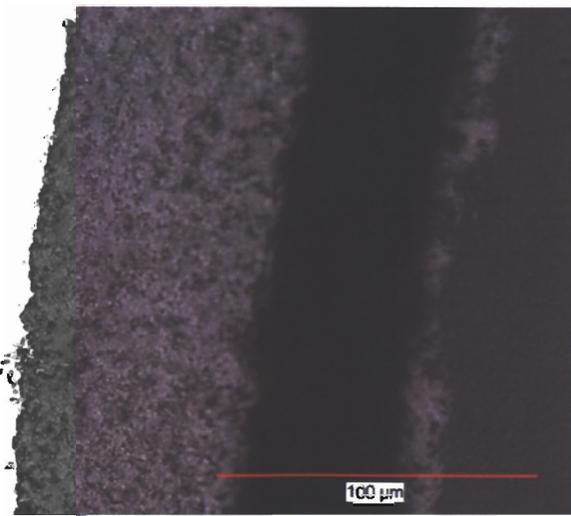


200x



500x

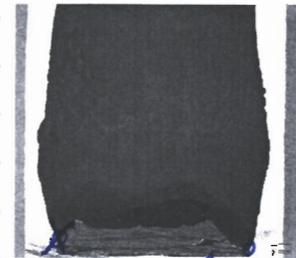
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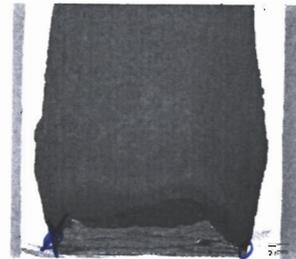
16 July 2007 M.P.



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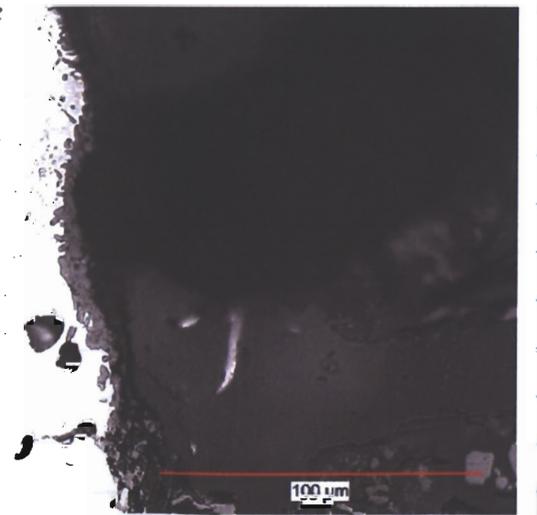
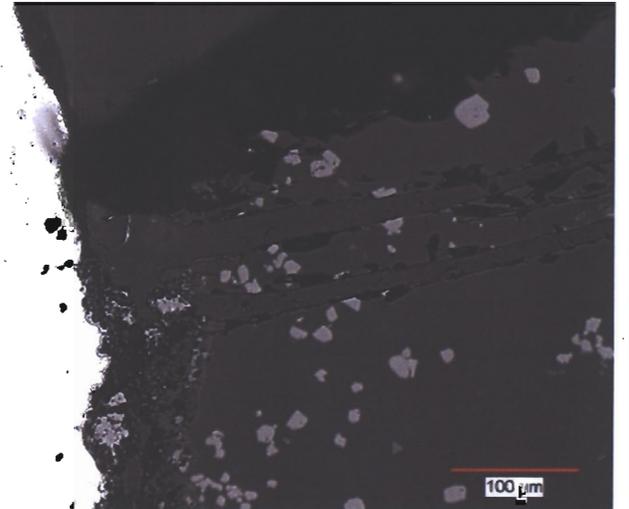
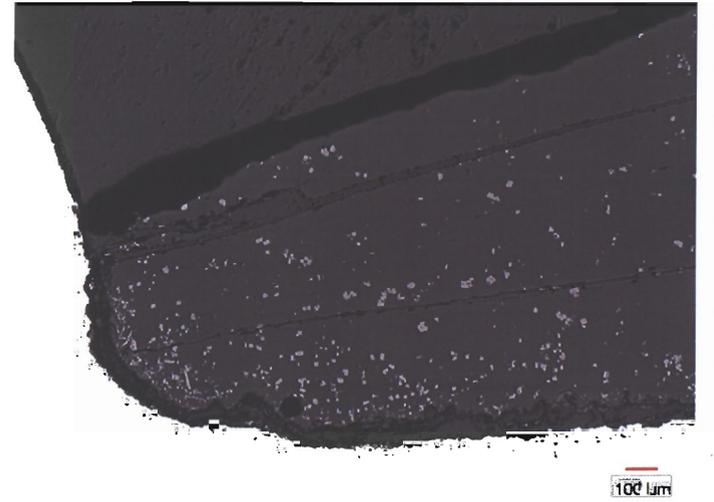


200x



500x

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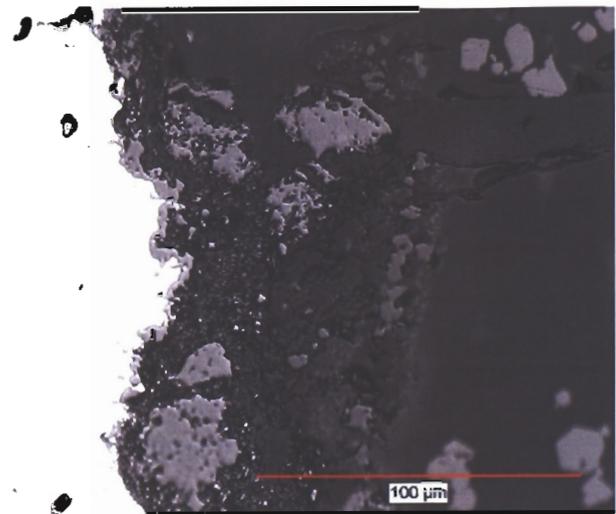


16 July 2007 M.H.

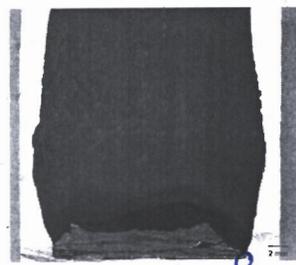
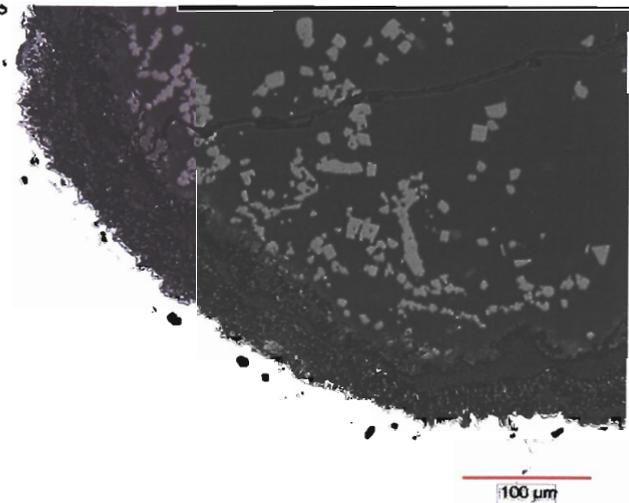


MP 7/16/07

500x
Slightly lower than
previous image

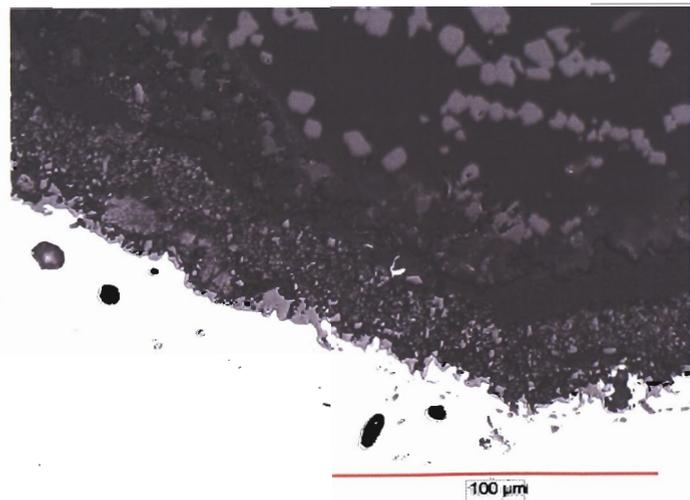


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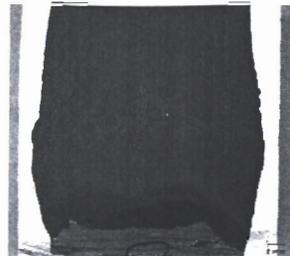


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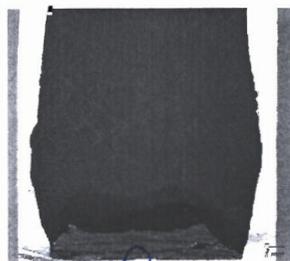
K.T. Chiang
8/15/07



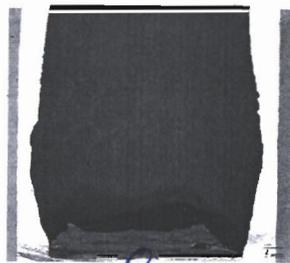
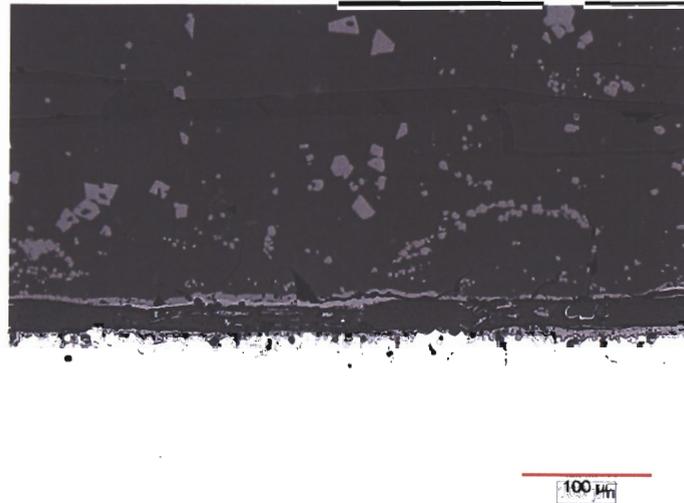
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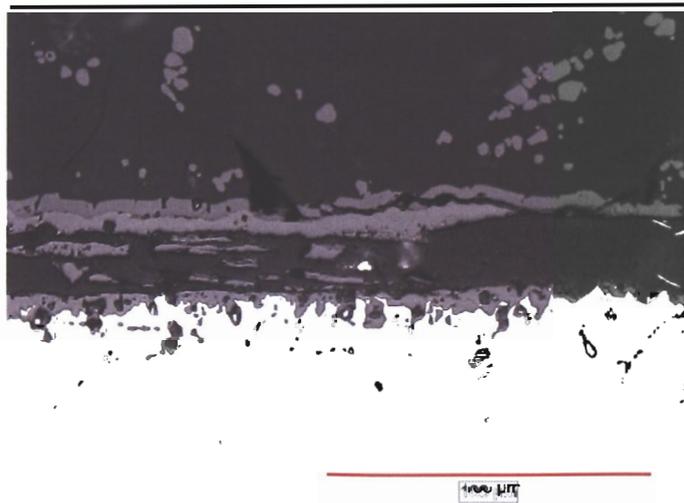


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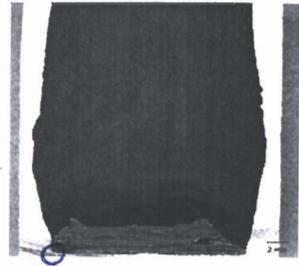


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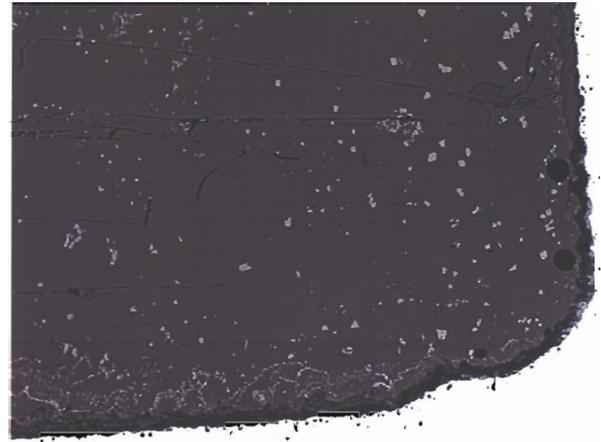
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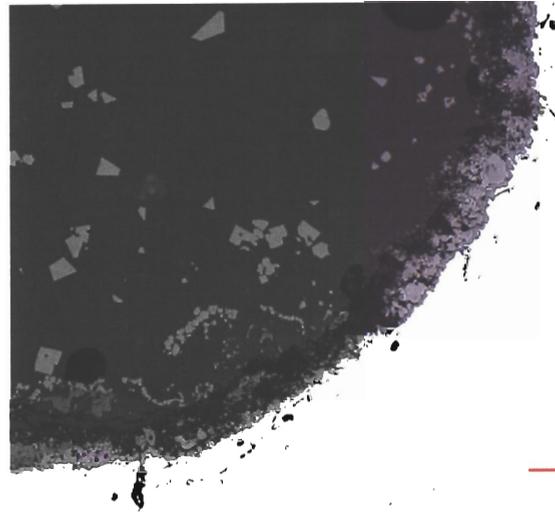
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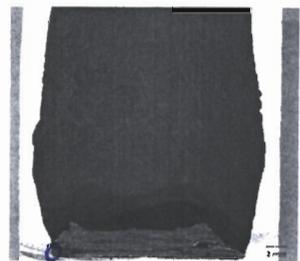
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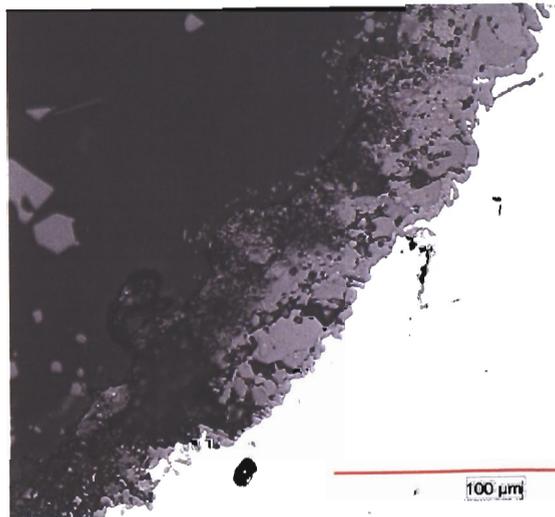
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100 μm



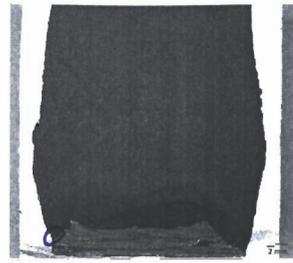
500x



100 μm

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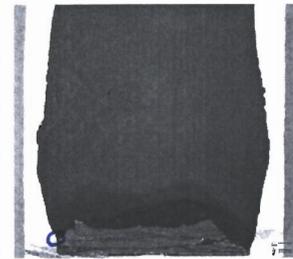
16 July 2007 M.P.



50x



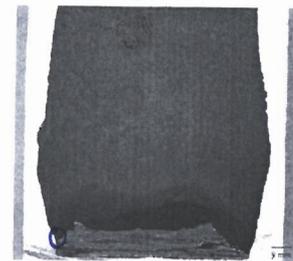
100 μm



200x
Bottom of Triple
Point



100 μm



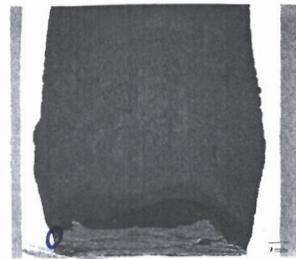
500x
Bottom of Triple
Point



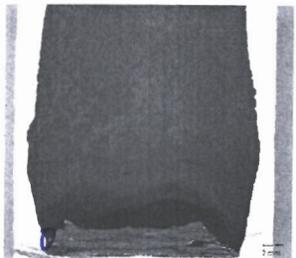
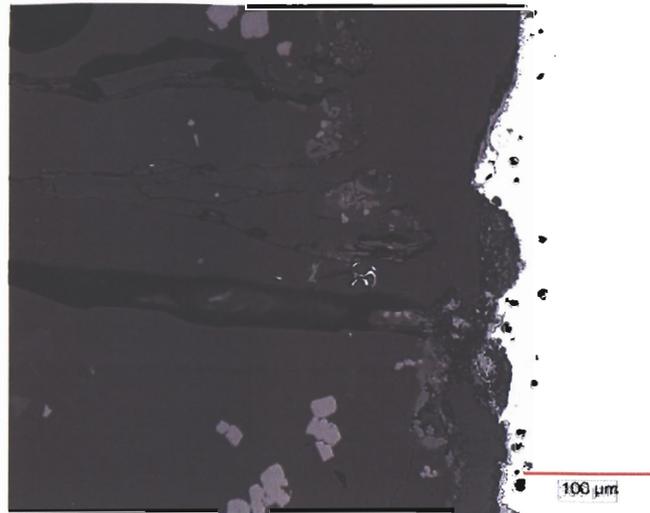
100 μm

K. J. Chief
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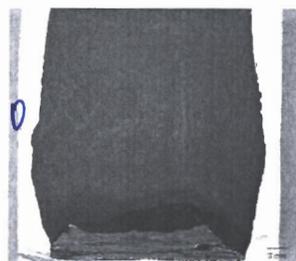
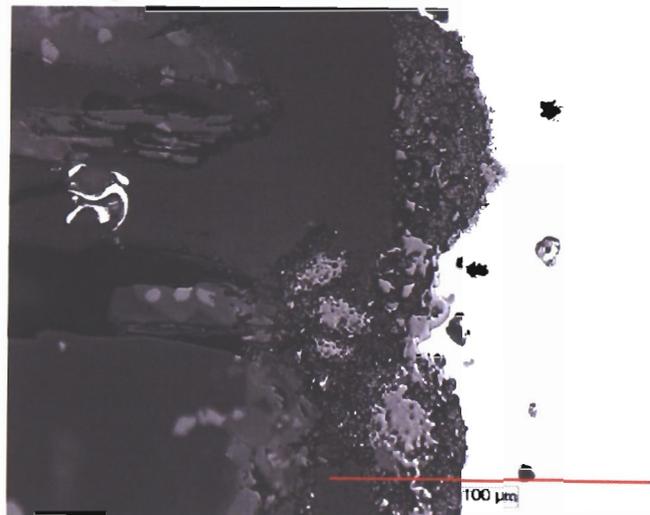
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200x
Just above Triple
Point



500x
Just above Triple
Point

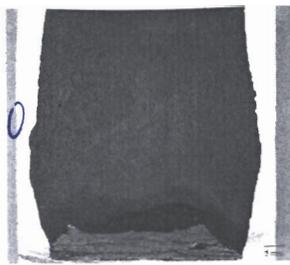


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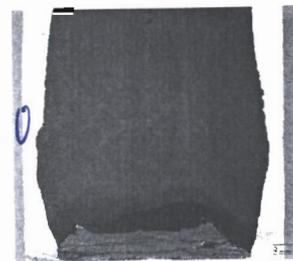
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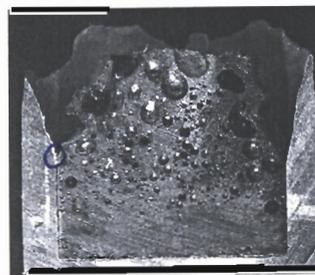
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500x

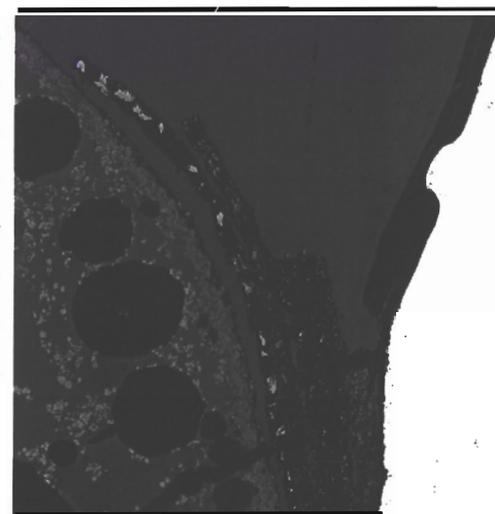


LW 4-4 Crucible
Temperature: 1180°C
Environment: Air
Time: 24 hr.

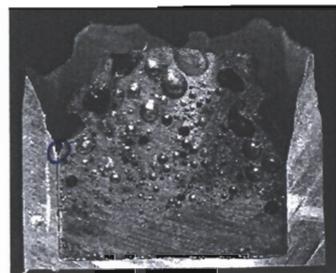


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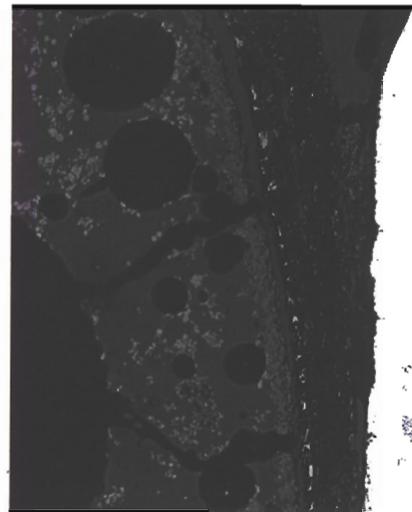
K.J. Chiraf
8/15/07



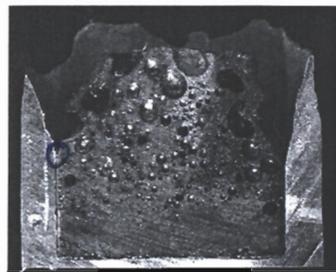
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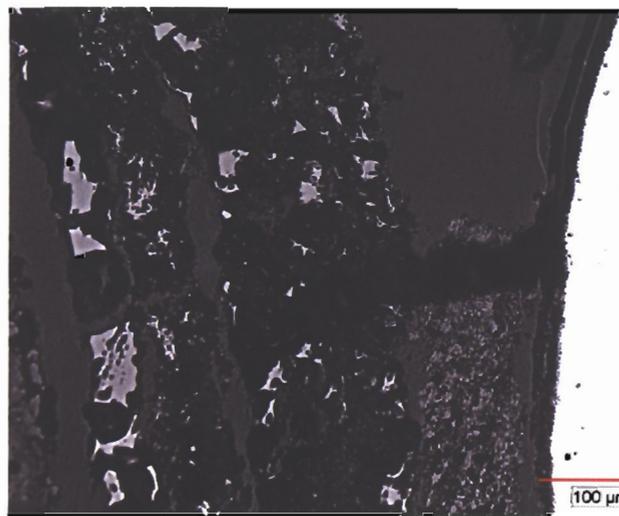
50x
slightly lower
than previous image



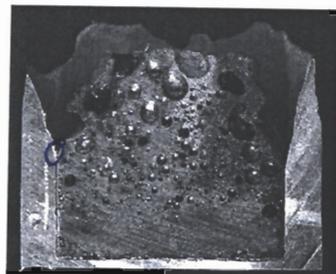
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200x

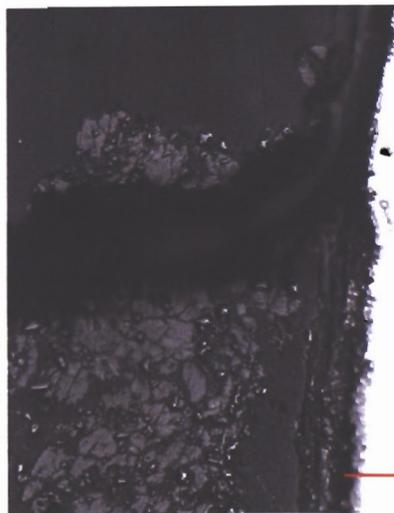


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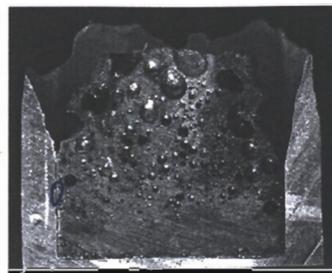
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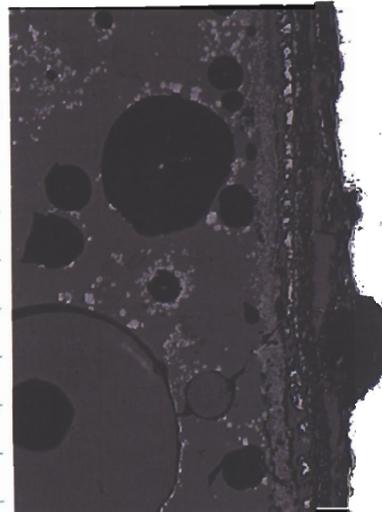


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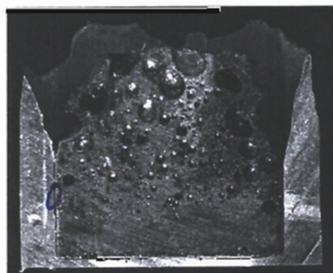
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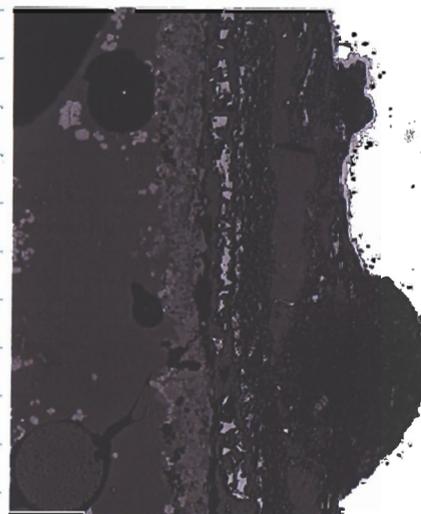
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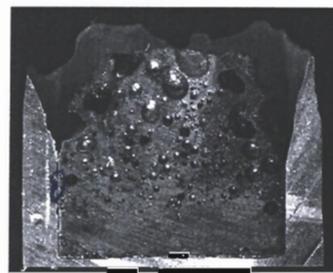
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100x



100 μm



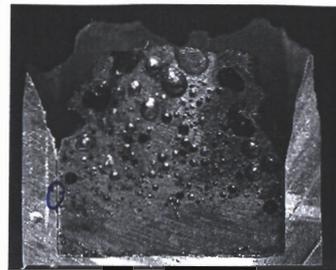
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100 μm

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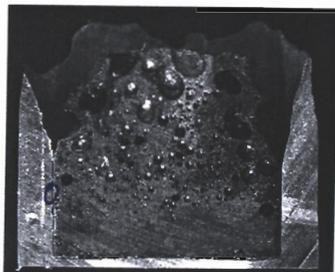
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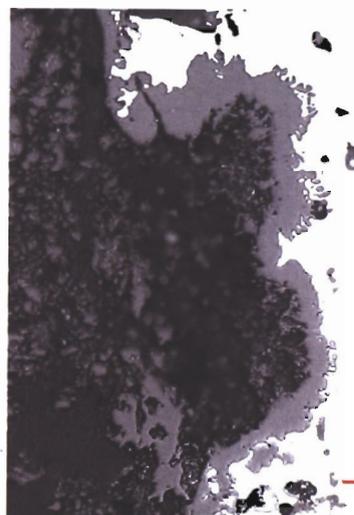
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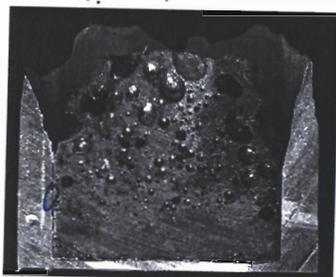
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500X



100 μm



500X

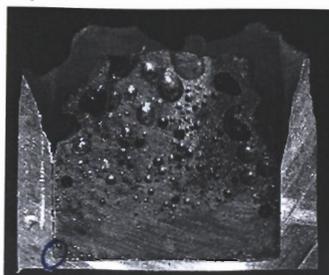


100 μm

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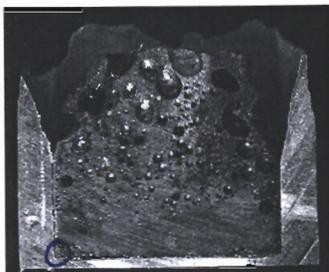
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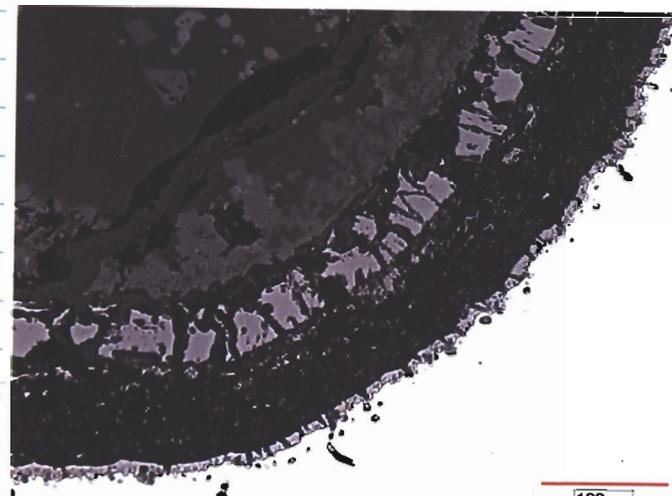
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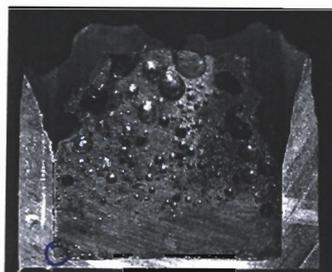
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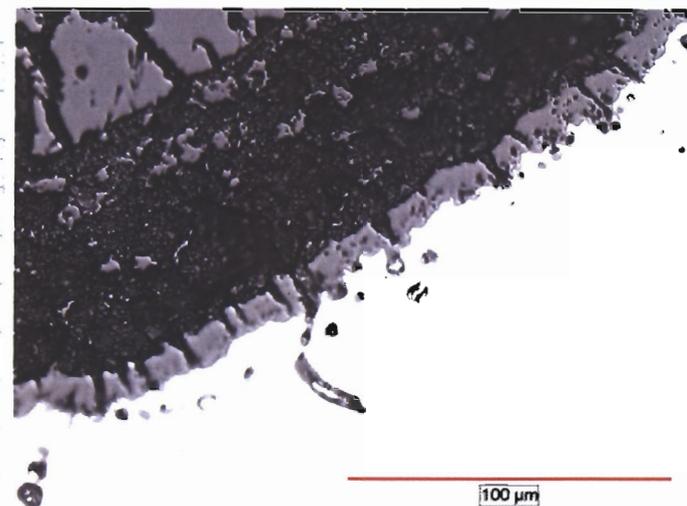
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100 μm



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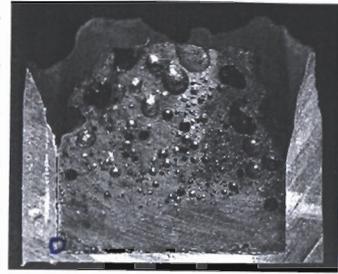


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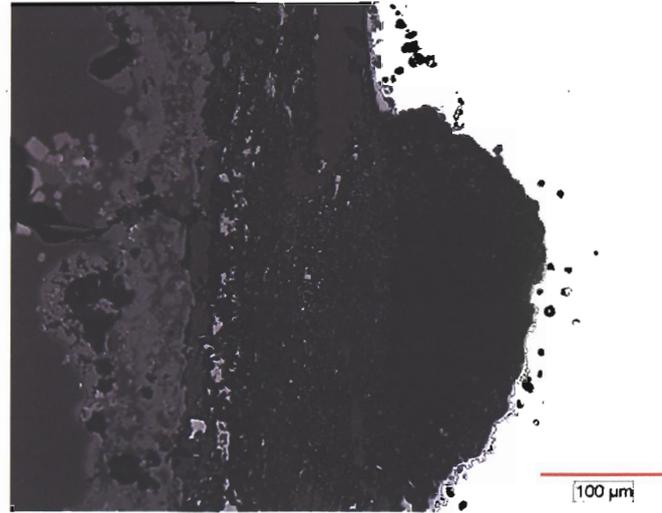
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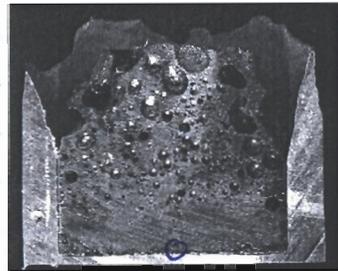
16 July 2007 M.P.



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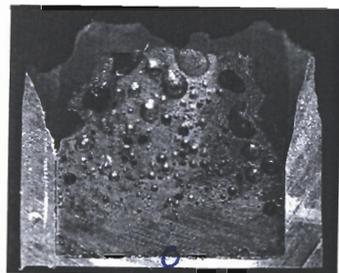
100 μm



50x



100 μm



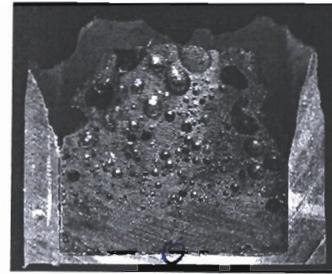
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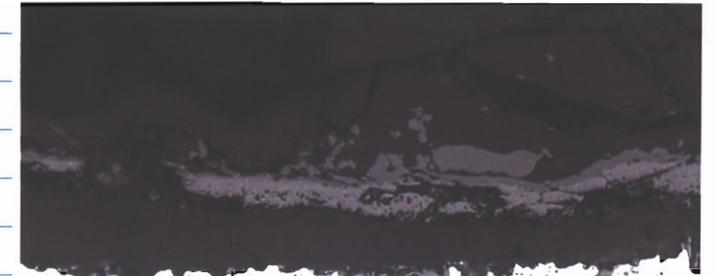
100 μm

K. T. Chiang 8/15/07

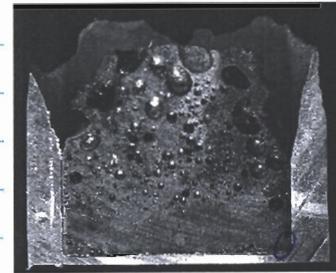
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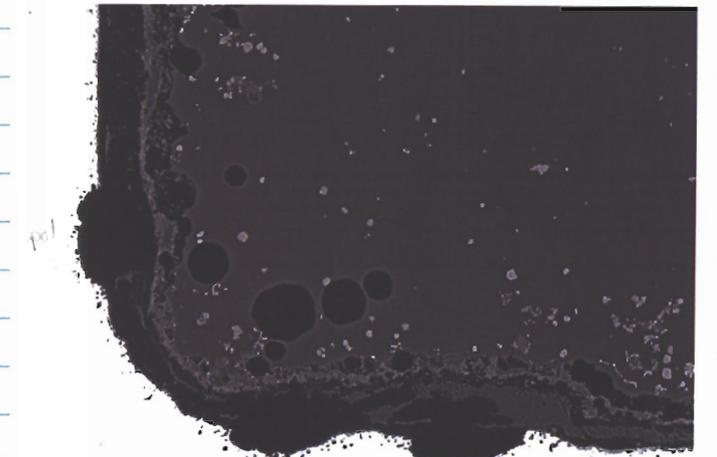
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100 μm



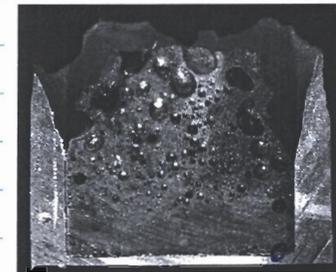
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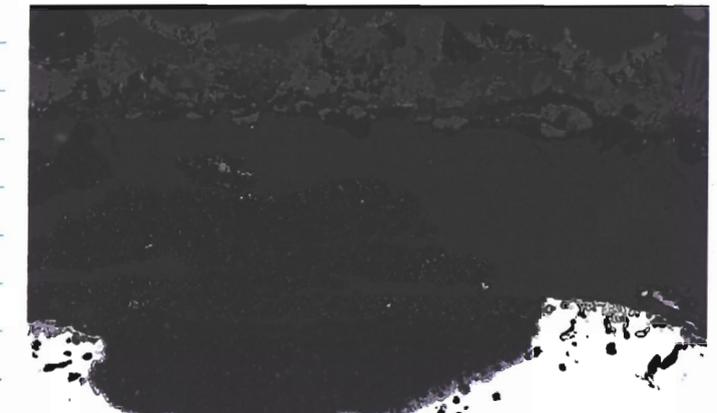
100 μm

pot 2

Spot 1



200x

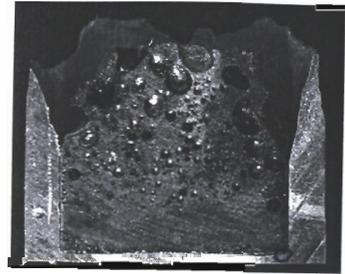


100 μm

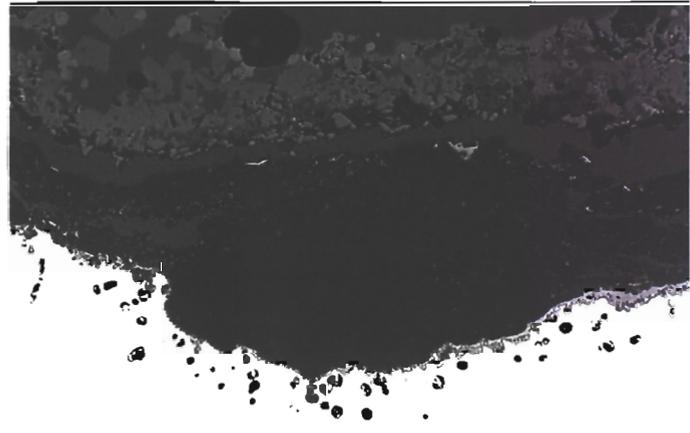
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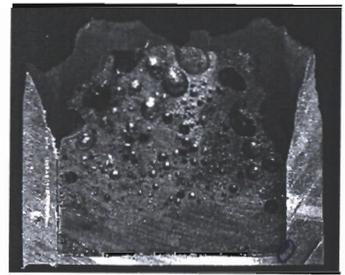
16 July 2007 M.P.



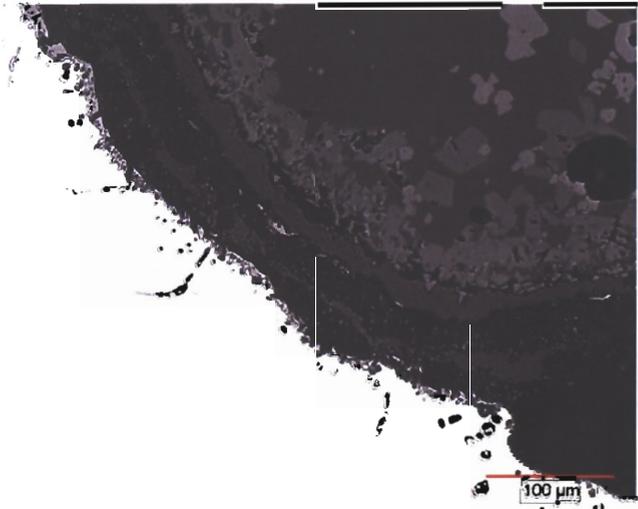
200x



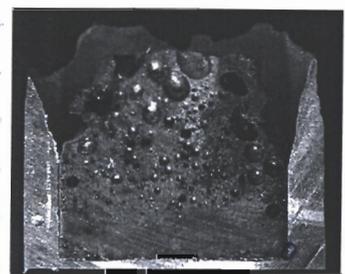
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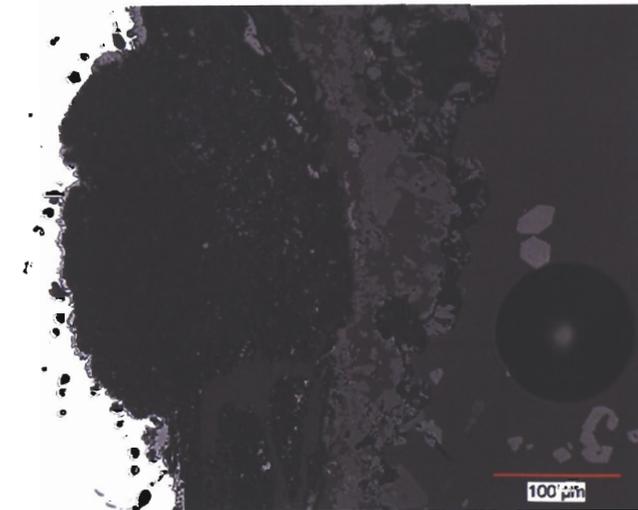
200x



100 μm



200x

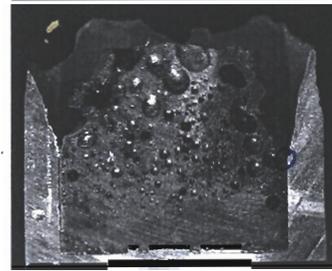


100 μm

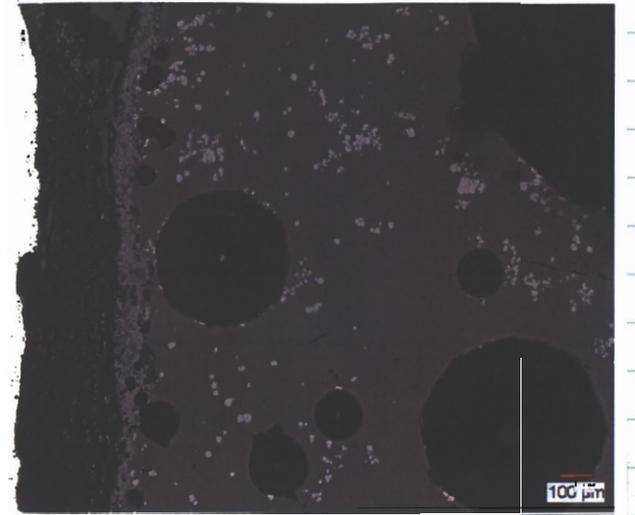
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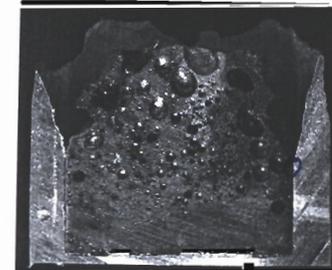
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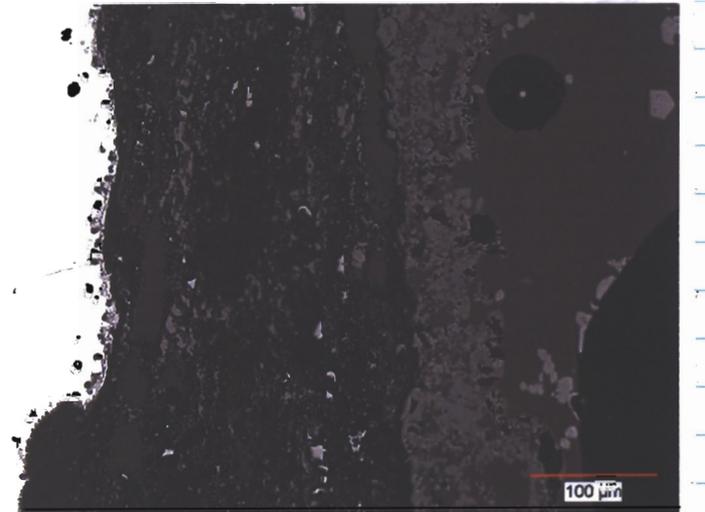
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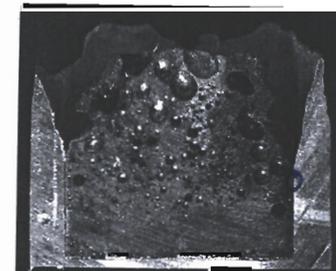
100 μm



200x



100 μm



200x



100 μm

K. J. Chiang

8/15/07