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Scientific Notebook # 009: Material Characterization Program (IWPE)

SOUTHWEST RESEARCH INSTITUTE
6220 CULEBRA ROAD
POSTAL DRAWER 28510
SAN ANTONIO, TEXAS 78284

IWPE
MATERIALS CHARACTERIZATION
(MARCH 28, 1990 – )
N. SRIDHAR

CNWRA-009

21
150

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Material Characterization Program

Start Date: March 28, 1990

# INDEX

INDEX



INDEX

3/28/90 MICROSTRUCTURES OF AS-RECIEVED PLATES

( See page 89 for initial entry on purpose, equipment, personnel)

Samples from 0.5" plates of the following materials were cut for optical metallography and SEM-EDX analysis :

(1) AISI 304 L stainless : Heat No. : T0954 - Jorgensen
(2) AISI 316 L stainless : Heat No. : P80746 - Jorgensen
(3) INCOLOY 825 : Heat No. : HH4371FC - Metal Goods
(4) Hastelloy alloy C-22 : Heat No. : 2277-8-3175 - Metal Goods
(5) CDA-102 (OFHC) : Lot No. : 6681 - Revere Copper
(6) CDA-613 (Cu-Al) : Heat No : ~~7037~~ 3/28 M5459 - Ampco Metal
(7) CDA-715 (Cu-Ni) : Heat No : 7037 Lot No : 6132A - Revere Copper

- Longitudinal and Transeverse sections were cut from all pieces.
- SEM-EDX Analyses were conducted on the mill surface and in the middle of the thickness cross-section (as baseline).
- The analysis was standardless. But the mid-section analysis provided an internal standard because it can be compared to the heat analysis supplied by the vendors.
- Optical metallography was performed on all the pieces.

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3/28/90



3/28/90 SEM-EDX Analysis of Hastelloy alloy C-22

Heat 2277-8-3175

| Mill Surface | Analysis 1 Wt% | Analysis 2 Wt% |
| --- | --- | --- |
| Ni (Kα) | 62.13 | 61.84 |
| Cr (Kα) | 13.63 | 13.19 |
| Mo (Lα) | 12.98 | 13.02 |
| W (M) | 4.63 | 5.12 |
| Fe (Kα) | 4.03 | 4.25 |
| Co (Kα) | 1.11 | 1.25 |
| Al (Kα) | 1.50 | 1.33 |

| Mid-section Analysis | Analysis 1 Wt% | Analysis 2 Wt% |
| --- | --- | --- |
| Ni | 57.69 | 58.05 |
| Cr | 21.43 | 21.32 |
| Mo | 11.32 | 11.52 |
| W | 4.20 | 3.75 |
| Fe | 4.16 | 4.19 |
| Co | 0.52 | 0.61 |
| Al | 0.68 | 0.55 |

Analysis supplied by vendor (Non-SEM Technique)

| | |
| --- | --- |
| Ni | Balance |
| Cr | 21.4 |
| Mo | 13.6 |
| W | 3.0 |
| Fe | 3.8 |
| Co | 0.89 |
| Al | N/A |

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## SEM Analysis of Incoloy alloy 825

Heat : HH 4371 FC

Mill surface

| | Wt% |
|---|---|
| Fe (K) | 43.46 |
| Ni (K) | 35.29 |
| Cr (K) | 15.77 |
| Ti (K) | 0.40 |
| Cu (K) | 1.55 |
| Mo (L) | 2.06 |
| Si (K) | 0.93 |
| Al (K) | 0.54 |

Mid-section

| | |
|---|---|
| Fe | 28.97 |
| Ni | 42.00 |
| Cr | 22.7 |
| Ti | 0.91 |
| Cu | 1.54 |
| Mo | 3.16 |
| Si | 0.54 |
| Al | 0.18 |

Vendor Analysis (Non-SEM Technique)

| | |
|---|---|
| Fe | 30.41 |
| Ni | 41.06 |
| Cr | 22.09 |
| Ti | 0.82 |
| Cu | 1.79 |
| Mo | 3.21 |
| Si | 0.19 |
| Al | 0.07 |

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3/28/90

## SEM Analysis of 304L stainless steel

Heat No. T0954

Mill Surface

| | Wt% |
|---|---|
| Fe (K) | 68.73 |
| Ni (K) | 8.92 |
| Cr (K) | 18.99 |
| Si (K) | 1.17 |
| Al (K) | 0.37 |
| Mn (K) | 1.57 |
| Cu (K) | 0.26 |

Mid-section

| | |
|---|---|
| Fe | 69.22 |
| Ni | 8.89 |
| Cr | 18.59 |
| Si | 1.49 |
| Al | 0.17 |
| Mn | 1.37 |
| Cu | 0.27 |

Vendor Analysis (Non-SEM Technique)

| | |
|---|---|
| Fe | Balance |
| Ni | 9.14 |
| Cr | 18.27 |
| Si | 0.47 |
| Al | N/A |
| Mn | 1.46 |
| Cu | N/A |

N. Sridhar
3/28/90

3/28/90 SEM Analyses of 316L stainless steel

Heat No. : P80746.

**Mill surface**

| | wt%. |
|---|---|
| Fe | 68.52 |
| Ni | 9.36 |
| Cr | 16.92 |
| Si | 0.94 |
| Al | 0.29 |
| Mn | ~~[illegible]~~ 1.99 (NS 3/28/90) |
| Cu | 0.25 |
| Mo | 1.74 |

**Mid-section**

| | |
|---|---|
| Fe | 68.51 |
| Ni | 9.36 |
| Cr | 16.92 |
| Si | 0.94 |
| Al | 0.29 |
| Mn | 1.99 |
| Cu | 0.25 |
| Mo | 1.74 |

Note: These numbers appear to be identical to the above numbers which is unlikely to occur in reality. The numbers should be checked in the original file. NS 3/28/90

**Vendor Analysis (Non-SEM Analysis)**

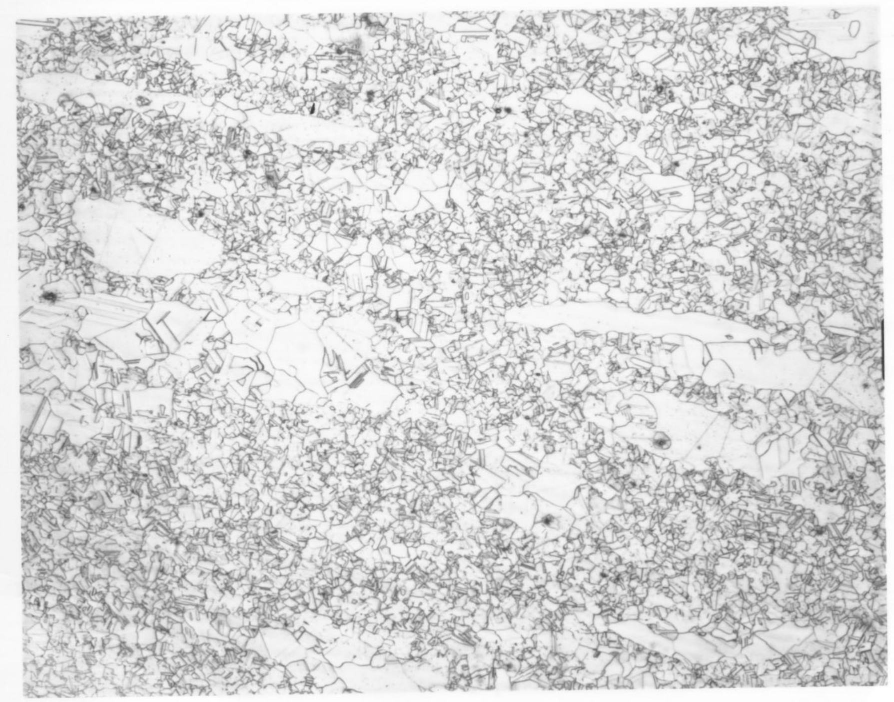
| | |
|---|---|
| Fe | Balance |
| Ni | 10.04 |
| Cr | 16.39 |
| Si | 0.49 |
| Al | N/A |
| Mn | 1.51 |
| Cu | 0.27 |
| Mo | 2.07 |

N. Sridhar
3/28/90



3/28/90 Photo micrograph of Incoloy alloy 825, 0.5" plate

– Same heat mentioned in page 4.



Transverse direction

Mag: 50X

Neg. #: 042235

3/27/90

Etch: HCl+Oxalic electrolytic.

Transverse

500X

Neg. # 041777

Etch: HCl+Oxalic electrolytic

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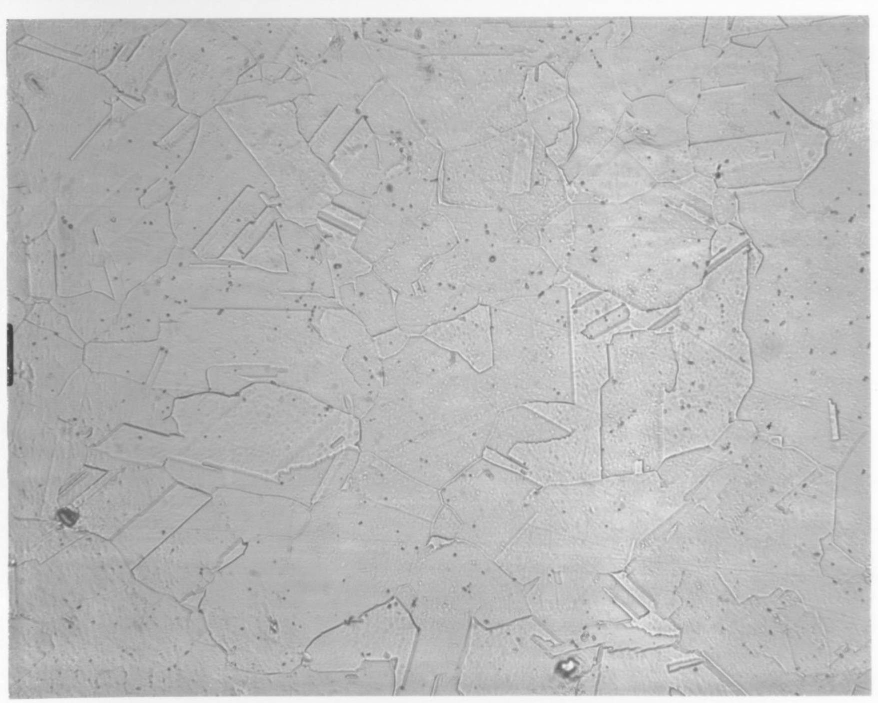
3/28/90 Photomicrographs of Incoloy alloy 825
(same heat as previous page)

200 X

Longitudinal

Neg. No : 41845



500X

Longitudinal

Neg. No. 41846

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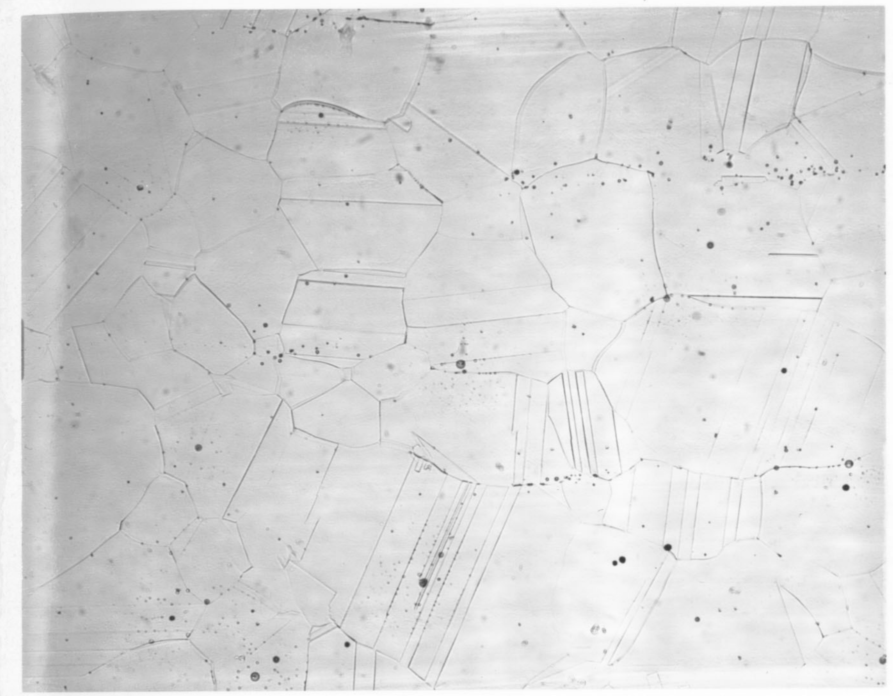
3/28/90



3/29/90 Photomicrographs of Hastelloy alloy C-22

same heat as before. Orientation unknown.



200 X

Neg. No. 041785

oxalic + HCl
etch.

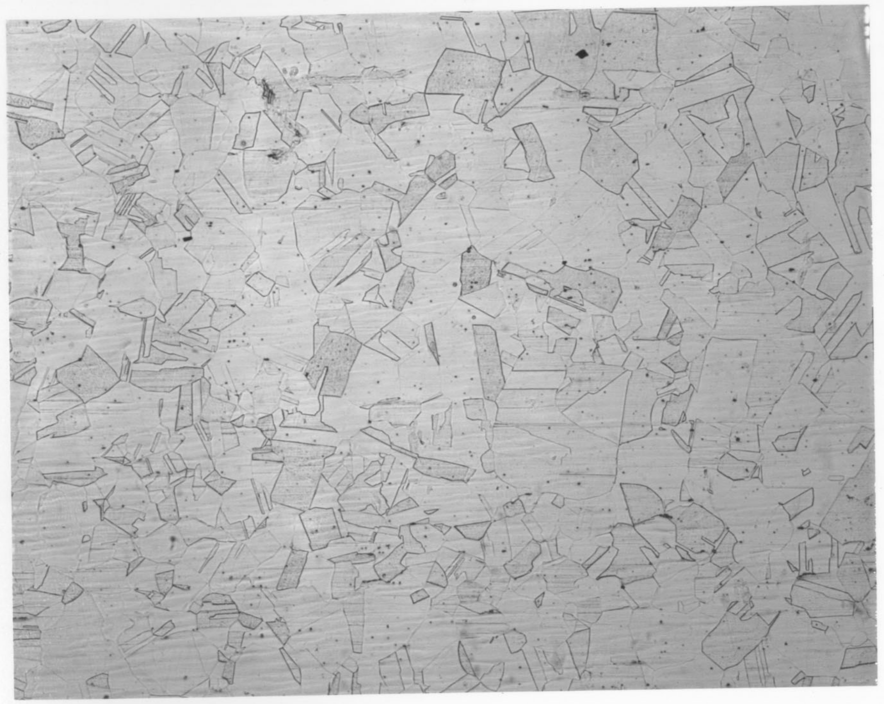
electrolytic

100X

Neg. No. 41784

N. Sridhar
3/29/90

3/29/90 Microstructures of 304L

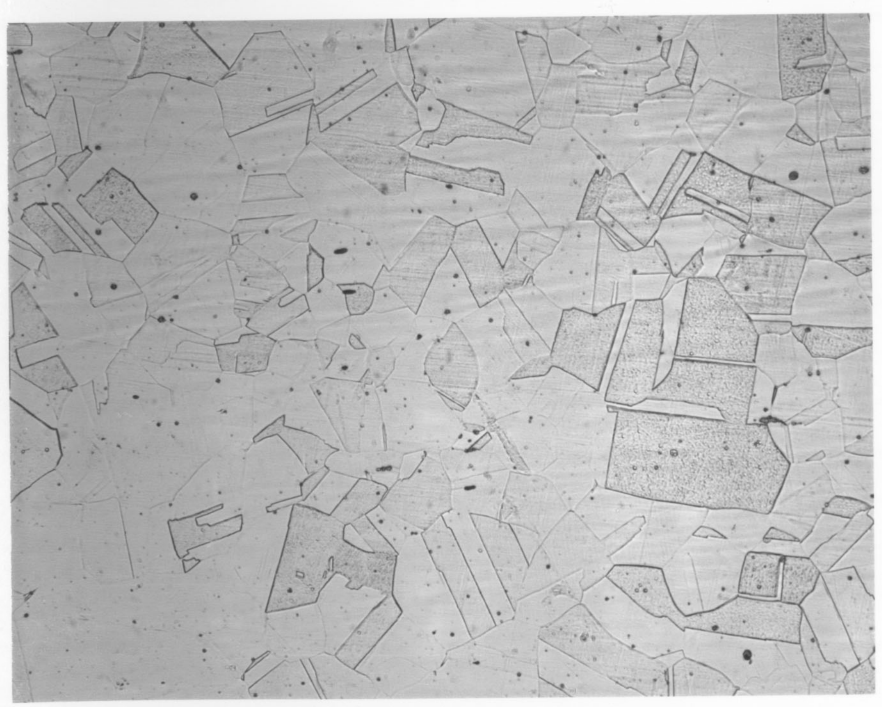


Transverse

100x

Neg. 041770

Oxalic acid etch.
electrolytic



Transverse

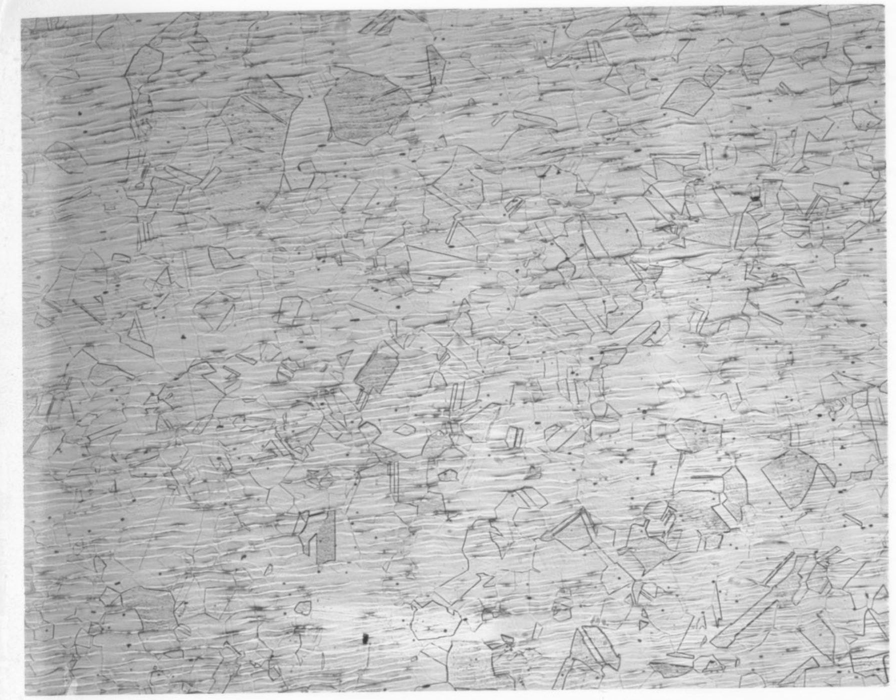
200x

Neg. No. 041771

N. Sridhar
3/29/90



3/29/90 Microstructure of 316L



Transverse

100x

Neg. No.
041778

oxalic acid
etch electrolytic

The lines in the microstructures are either artifacts from metallography or too heavy an etch of the heavily worked areas. The techniques are being modified to determine the suitable approach.

There are also non-metallic inclusions.

N. Sridhar
3/29/90

3/29/90 Microstructures of CDA 102 – OFHC.
(same lot as shown before)

Transverse

50x

Neg. 041838

$FeCl_3 + HCl$
etch.
(immersion)

Transverse

100x

Neg. 041840

N. Sridhar
3/29/90



3/29/90 Microstructures of CDA-102 (OFHC)
3/29/90

Longitudinal

50x

Neg. 041796

$FeCl_3 + HCl$
etch.
(immersion)

Longitudinal

100x

Neg. No. 041797

$FeCl_3 + HCl$
etch.

N. Sridhar
3/29/90

ASTM A262 Practice B on 304L SS

This test was to determine the susceptibility of the alloy to intergranular corrosion.

4 coupons of dimensions 1¼" × ¾" × ⅛" were cut from a plate of the heat no. T0954. The specimens were polished with 120 grit paper. Two were polished only on the cut edges, leaving the mill finish on the largest faces. Two were polished on all 6 faces.

The test solution and procedures were prepared and followed according to ASTM A262 Practice B.

The specimens were identified as follows:

304-1 (mill finish)
304-2 (mill finish)
304-3 (polished)
304-4 (polished)

| | Initial Wt | Final Wt | ΔWt |
|---|---|---|---|
| 304-1 | 55.2967 | 55.1183 | 0.1784 grams |
| 304-2 | 54.5766 | 54.3836 | 0.1930 " |
| 304-3 | 55.3729 | 55.2252 | 0.1477 " |
| 304-4 | 54.1446 | 53.9866 | 0.1580 " |

Corrosion rates were calculated according to:

$$\text{inches per month} = \frac{(287 \times W)}{(A \times t \times d)} \text{ ; ipm} \times 12000 = \text{mils/year}$$

W = Δwt ; A = cm² (area) ; t = time (hrs) d = density (7.9 gr/cm³)

| | | | |
|---|---|---|---|
| CR | 304-1 | 25.94 | mpy |
| " | 304-2 | 28.05 | " |
| " | 304-3 | 21.46 | " |
| " | 304-4 | 22.96 | " |

W Smachowski



ASTM A262 Practice B on 316L SS

Purpose: To determine susceptibility of 316L to intergranular corrosion.

Four coupons of dimensions 1¼ × ¾ × ⅛" were cut from a plate of Heat No. P80746. These were polished as described on p.16, such that two coupons had mill finish on two sides and two were polished on all sides.

All procedures and solution preparation were done in accordance with ASTM A262 Practice B

| | | Orig wt | Final wt. | ΔWt. |
|---|---|---|---|---|
| 316 #1 | (mill finish) | 61.1141 | 60.8397 | 0.2744 grams |
| 316 #2 | (" ") | 63.1016 | 62.8381 | 0.2635 " |
| 316 #3 | (polished) | 66.4753 | 66.2379 | 0.2374 " |
| 316 #4 | (polished) | 60.0887 | 59.8309 | 0.2578 " |

Corrosion rates were calculated using the formula on p.16. 8.0 g/cm³ was used as the density.

| | | | |
|---|---|---|---|
| C.R. | 316 #1 | 39.38 | mpy |
| " | 316 #2 | 37.81 | " |
| " | 316 #3 | 34.07 | " |
| " | 316 #4 | 36.99 | " |

N Sniffles
12/1/94

WJ Machowski
12/90

ASTM G28 Prac. A on Incoloy 825

Purpose: To determine susceptibility to intergranular corrosion.

Four specimens were cut from a plate of Heat No. HH 4371 FC. The dimensions and area are listed below. These were polished as described on p.16 so that two coupons retained the mill finish. Solutions and procedures were done in accordance with ASTM G-28 Practice A. Exposure was 120 hrs.

| | | | | | Area ($cm^2$) |
|---|---|---|---|---|---|
| 825 #1 | (mill finish) | 3.247 cm × | 1.613 cm × | 1.299 cm | 23.10 |
| 825 #2 | ( " " ) | 3.142 | × 1.539 | × 1.300 | 21.75 |
| 825 #3 | (polished) | 2.881 | × 1.648 | × 1.299 | 21.26 |
| 825 #4 | ( " ) | 2.982 | × 1.599 | × 1.299 | 21.44 |

| | Orig Wt | Final Wt. | ΔWt | |
|---|---|---|---|---|
| 825 #1 | 55.4028 | 55.3130 | 0.0898 | grams |
| 825 #2 | 50.9399 | 50.8471 | 0.0928 | " |
| 825 #3 | 50.0570 | 50.0273 | 0.0297 | " |
| 825 #4 | 49.4960 | 49.4660 | 0.0300 | " |

Corrosion rates in mils per year were calculated using the formula

$$C.R. = (K \times W)/(A * T \times D) \quad \text{where}$$

$K = 3.45 \times 10^6$ (constant for mpy) $W$ = weight loss
$A$ = area ($cm^2$) $T$ = time (hrs) $D$ = density ($g/cm^3$)
$D = 8.14$

| | | | |
|---|---|---|---|
| C.R. | 825 #1 | 13.73 | mpy |
| " | 825 #2 | 15.07 | " |
| " | 825 #3 | 4.93 | " |
| " | 825 #4 | 4.94 | " |

W J Machowski
5/4/90



ASTM G-28 Prac. A on Hast C-22

Purpose: To determine susceptibility of C-22 to intergranular corrosion.

Four specimens of C-22 were cut from a plate of Heat No. 2277-8-3175. These were polished as described on p.16 so that two coupons retained their mill finish. Solution preparation and procedures were done in accordance with ASTM G-28 Practice A.

| | | | | | Area ($cm^2$) |
|---|---|---|---|---|---|
| C-22 #1 | (mill finish) | 3.095 cm × | 1.889 cm × | 1.272 cm | 24.37 |
| C-22 #2 | ( " " ) | 3.082 | × 1.925 | × 1.270 | 24.58 |
| C-22 #3 | (polished) | 2.982 | × 2.149 | × 1.262 | 25.77 |
| C-22 #4 | ( " ) | 2.983 | 1.865 | 1.261 | 23.35 |

| | Orig. Wt | Final Wt | ΔWt | |
|---|---|---|---|---|
| C-22 #1 | 64.4996 | 64.4025 | 0.0971 | grams |
| C-22 #2 | 65.8977 | 65.7986 | 0.0991 | " |
| C-22 #3 | 69.6989 | 69.6543 | 0.0446 | " |
| C-22 #4 | 61.0789 | 61.0391 | 0.0398 | " |

Exposure was for 24 hours. Corrosion rates were calculated using the formula on p. 18.
For C-22, D = 8.60

| | | | |
|---|---|---|---|
| C.R. | C-22 #1 | 66.60 | mpy |
| " | C-22 #2 | 67.39 | " |
| " | C-22 #3 | 28.93 | " |
| " | C-22 #4 | 28.49 | " |

W J Machowski
5/4/90

ASTM G-28 Practice B on Hast C-22

Purpose: To determine the susceptibility of C-22 to intergranular corrosion.

Four specimens of C-22 were cut from a plate of Heat No. 2277-8-3175. These were polished as described on p.16 so that two coupons retained their mill finish. Solution preparation and procedures were done in accordance with ASTM G-28 Practice B.

| | | | | | $A(cm^2)$ |
|---|---|---|---|---|---|
| C-22 #5 | (mill finish) | 3.072 cm | × 1.819 cm | × 1.273 | 23.63 |
| C-22 #6 | ( " " ) | 3.055 | × 2.018 | × 1.269 | 25.21 |
| C-22 #7 | (polished) | 2.938 | 1.936 | 1.263 | 23.69 |
| C-22 #8 | ( " ) | 2.950 | 1.782 | 1.262 | 22.46 |

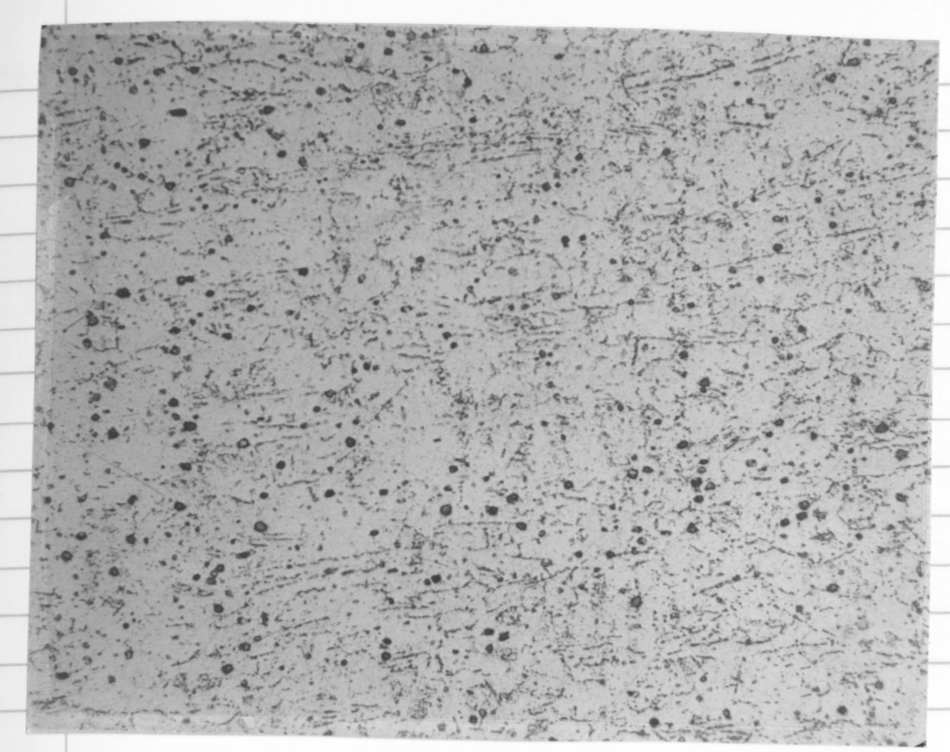
| | Orig. Wt. | Final Wt | ΔWt | |
|---|---|---|---|---|
| C-22 #5 | 61.5789 | 61.5440 | 0.0349 | grams |
| C-22 #6 | 67.0563 | 67.0195 | 0.0368 | " |
| C-22 #7 | 62.8499 | 62.8419 | 0.0080 | " |
| C-22 #8 | 57.7277 | 57.7202 | 0.0075 | " |

Exposure was for 24 hours. Corrosion rates were calculated using the formula on p.18. For C-22, D=8.69

| | | | |
|---|---|---|---|
| C.R. | C-22 #5 | 24.69 mpy | av. 24.55 |
| " | C-22 #6 | 24.40 " | |
| " | C-22 #7 | 5.64 " | 5.61 |
| " | C-22 #8 | 5.58 | |

W J Machowski
5/10/90





500X
$FeCl_3$ etch
Neg. 42478

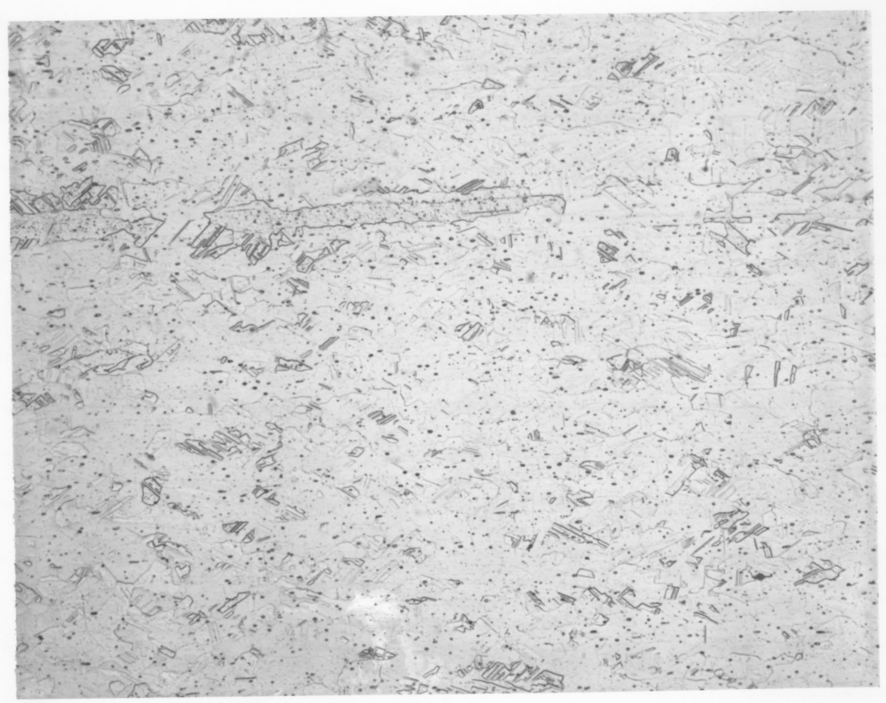
CDA-613, As-received 0.5" Plate. Longitudinal Section

500X
$FeCl_3$ etch
Neg. 42481

CDA-613, As-recieved 0.5" Plate. Transverse Section.

5/22/90 [illegible]

CDA 613. Reanneal 1500F, 30min, WQ. 500X. Neg. 42878. Long. Section



CDA 613 Reannealed 1500F, 30min, WQ. 500X
$FeCl_3$ etch. Transverse Section. Neg. 428789



5/28/90 SEM-EDX Analysis of as-received 0.5" plate of CDA-613 (Heat # M5459-AmpCo Metal). See page 4 for more details)

**Purpose:** Microstructures taken optically are shown in page 21. They exhibit a number of dark particles in the matrix. The purpose of these analyses was to get a chemical composition of these particles, at least, semi-quantitatively.

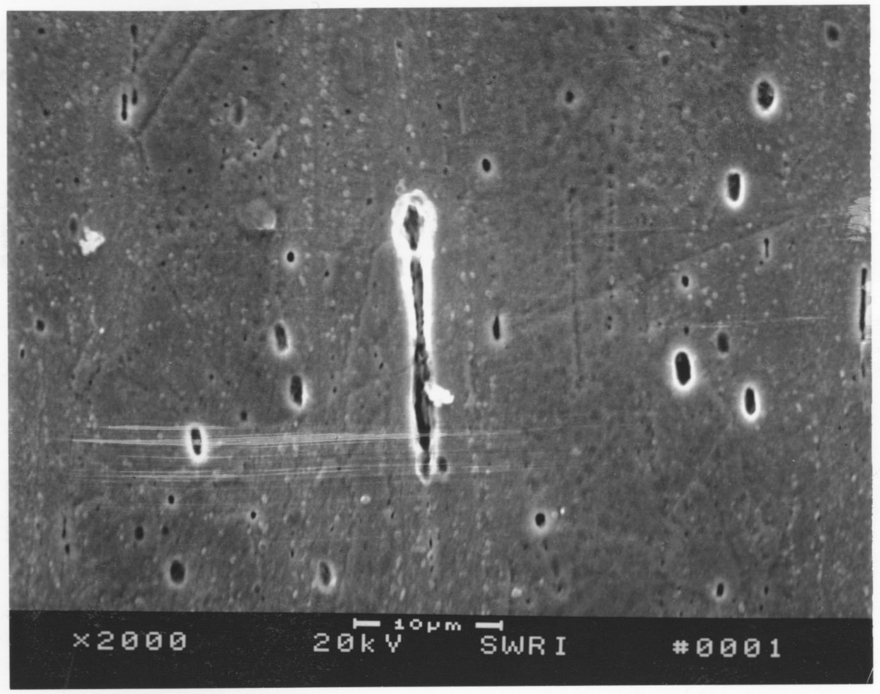
**Method:**
1) Mounted sample in bakelite, polish & etch.
2) ~~Sprns~~ Vapor deposited carbon on top to increase conductivity without interfering with SEM analysis.
3) SEM-EDX spot analyses of particles, spot analysis of matrix and overall area analysis of sample.
4) ZAF semi-quant. calculation of composition

**Results:** Data are shown in the next two pages.

**Conclusion:** The matrix particles appear to be rich in iron and low in Al. Reference to ASM Handbook suggests that such iron rich particles may be present in α-Cu-Al bronzes that contain iron.

5/28/90 N. Sridhar

5/28/90 SEM Images of areas Examined. CDA-613 (Same as previous page).




Neg. No. 42954

Particle No. 1.

CDA - 613


Neg. No. 42955

Particle No. 2

CDA - 613

N. Sridhar
5/28/90



5/28/90 Standardless Analysis of CDA-613 as-received plate Sample

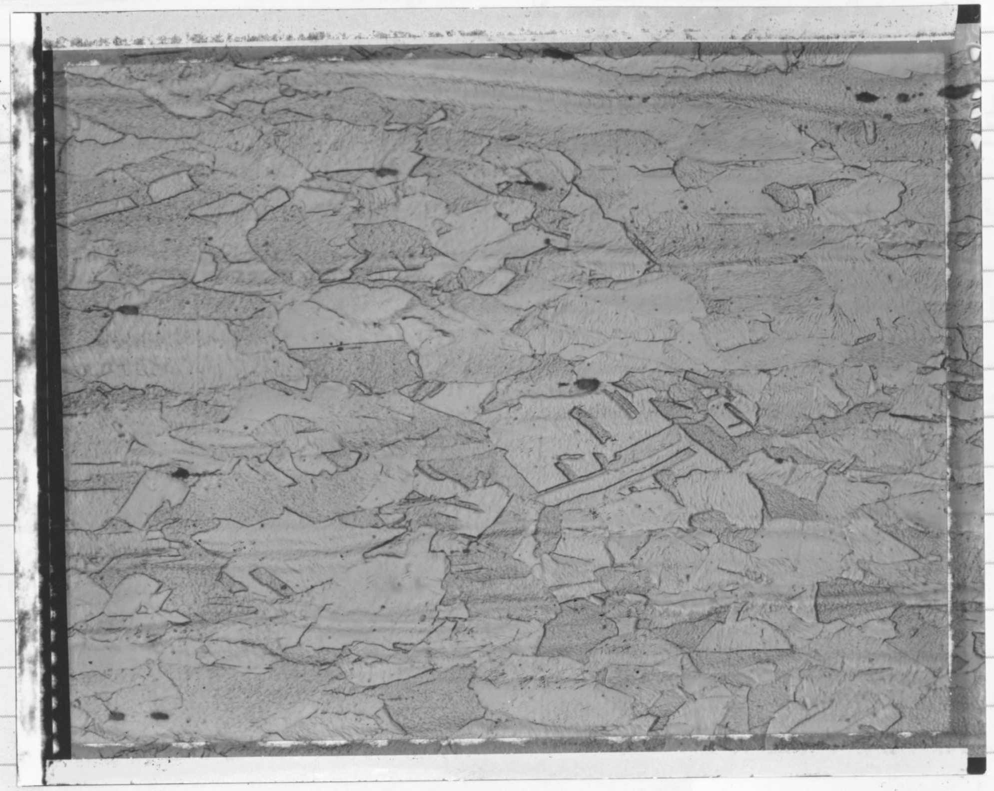
| Element | Overall Matrix | Spot Matrix | Elongated Particle 1 | Elongated Particle 2 | Heat Analysis Supplied by manufacturer |
|---|---|---|---|---|---|
| Cu | 92.2 | 92.4 | 91.5 | 61.4 | 90.7 |
| Al | 5.6 | 6.01 | 2.6 | 3.4 | 6.67 |
| Si | 0.33 | 0.34 | 0.24 | 0.28 | NIL |
| Fe | 1.94 | 1.30 | 5.68 | 33.25 | 2.35 |
| Ti | NP* | NP* | NP* | 0.68 | NR** |
| V | NP | NP* | NP* | 0.99 | NR |

* NP - Not Present in significant amounts for EDX Analysis
** NR - Not reported.

Elongated particle No. 1 may not have shown high Fe because the beam may not have been squarely on the particle or part of the particle may have fallen off.

14/1/94

N. Sridhar
5/28/90



CDA - 715, as-received plate, Neg. 59905, 500x

N. [illegible]
12/1/94



8/13/93

Metallography Technique for Alloy 825

This metallographical technique is for use on alloy 825. This technique will show any chromium depleted zones that might exist in the sample. Usually chromium depleted zones will be at grain boundaries where chromium carbides are formed or where an area of sigma phase exists. These zones will be visible when viewed with a microscope that is capable of at least X200 using reflected light. The zones will have a ditch-like structure and non depleted zones will have a step structure.

Grinding

The specimen must be brought to a 600 grit finish. If the sample has a thick scale or oxide layer on it but the surface is at or almost a 600 grit finish, the scale/oxide can be wet ground off with 600 grit paper at a wheel speed of 120 rpm. If the sample is not smooth, then the sample must be wet ground starting at 180 or 240 grit (whichever is available) at a wheel speed of 120 rpm. A wet grind with 320 grit at 120 rpm should be followed by 400 grit. A 600 grit wet grind should be the next. Try not to apply too much pressure to the sample when grinding. Too much pressure will put deep scratches in the sample that will take a long time to take out. All of the scratches for a given grain size should go the same way, this will help eliminate pitting of the sample. This is done by moving the sample slowly from the edge of the wheel, to the center and back to the edge. When coming to the edge of the wheel do not go off of the wheel, this will put bad scratches on the sample. All the edges that are on the polished face of the sample should be bevelled. If the sample is mounted, then the mount should be beveled.

If faceting the sample is a problem during the pre-600 grit stages, it is allowable to go from 180 grit directly to 600 grit. This will take longer but it is less likely that facets will occur. When doing this, start grinding with 600 grit until at least 50% of the sample has a 600 grit finish. At this time turn the sample 90°, on the polishing face, and continue polishing until all of the 600 grit scratches are going the same direction. When this is done repeat until all of the sample is at 600 grit. It will probably be necessary to replace the sanding disks many times before the sample is done. The disk is ready to be replaced when the scratches are finer than the original scratches. The disk can also be felt to see if there is any grit left on it.

Samples that are mounted and are too tall will be easy to facet. This can be somewhat corrected by making a shorter mount. However very short mounts are hard to control and allow fingertips to become ground.

Rough Polishing

When the sample is brought to a 600 grit finish the sample should be ultrasonicly cleaned with detergent for at least 5 minutes to remove any abrasive particles that could be sticking to the surface of the sample. Once done the sample should be rinsed off well. The sample is then polished on $3\mu$ diamond lapping film using $18M\Omega$ water + detergent as lubricant. The sample should be polished perpendicular to the previous scratches to insure that all the previous scratches are removed. Do not put too much pressure on the sample since deep scratches can

8/13/93

occur. If there are bubbles under the lapping film very deep scratches will occur. They can be found by feeling as if the sample has hit a "bump". The sample may or may not be planar from the 600 grit stage so the technique in the previous paragraph (adapted to $3\mu$ diamond film) may be needed to get the entire surface planar.

The lapping film will leave larger scratches than the diamond size of the film. Since this is true, the $3\mu$ lapping film doesn't leave a $3\mu$ finish. Therefore $3\mu$ paste should be used to receive a $3\mu$ finish. The reasons that the film is used are to make the sample planar, get a more uniform polish, decrease the amount of time required to polish the sample, and to cut down on the use of diamond paste. Along with being expensive, diamond paste will tend to leave pits in the sample the longer it's used. Diamond film will also last longer than diamond paste will.

When done with the $3\mu$ diamond lapping film, the sample should be rinsed off with water. It may be a good idea to swab off the surface. The sample should then be polished with $3\mu$ diamond paste on a polishing cloth using universal diamond extender as lubrication. The polishing cloths should be either Texmet (Buehler) or Pellon (PSI Testing Systems). Pellon is preferred because the cloth seems to last longer, however at the time that this was written, PSI had not overcome the problem of universal diamond extender dissolving the adhesive backing on the cloth. The cloth should be moist to slightly wet with extender. Diamond paste is syringed onto the cloth, approximately .25 to .5 grams, every other time the cloth is used. If the cloth is new it should be moistened with 18MΩ water after it is applied and more diamond paste should be used. The wheel should be at 120rpm. The sample should be polished perpendicular to the original scratches. This shouldn't take very long unless the lapping film scratches are deep. Periodic viewing can be done by rinsing off the sample of the surface of the specimen with 18MΩ water or diamond extender. All of the original scratches are gone when the surface looks hazy in one or two directions. When all of the original scratches are gone wash off with water and swab the surface well to remove any diamond particles that may be stuck to the surface. The sample should then be put into an ultrasonic bath for 5 minutes. The sample should be rinsed off with 18MΩ water.

## Final Polishing

The sample is then polished with Mastermet, colloidal silica alcohol solution, using either Microcloth (Buehler) or Final Polish (PSI Testing Systems) polishing cloths. The Mastermet solution is it's own lubricant. The wheel speed should be 120rpm. The Mastermet solution is shaken to make the solution homogeneous. Four disposable pipet's worth of solution is dispensed on the cloth and another is held on the side. If this is the fist use for the cloth, it should be moistened with 18MΩ water. Polish the sample in a rotating motion, counter direction of the wheel is best, until most of the Mastermet on the wheel is gone. Replenish the supply with the last pipet-full and continue to polish until almost gone. Again do not use too much pressure on the sample. Swab off the sample surface very well. It is believed that there are surfactants in Mastermet and they should be removed. It can be seen that they are removed when the water beads off the surface. The sample should be rinsed off with 18MΩ water. Check the sample after washing the surface, it is best to look under the microscope, to verify that all the scratches are gone. If the sample is not good enough repeat the procedure until the sample is adequate. When done polishing with the cloth, it is recommended to apply 18MΩ



8/13/93

water directly to the cloth to keep it moist. If the cloth dries out it will become ineffective or be damaging to the sample if reused. If the cloth is kept wet after the initial use, it can be used many times.

## Electro-Polishing

After checking the sample it should be immersed in Methanol immediately to prevent contamination on the sample surface. There should be two containers of solution, one etching and one polishing solution. The polishing solution is 170 ml Methanol + 30 ml 96% $H_2SO_4$. The etching solution consists of 190 ml of 36% HCL and 10g Oxalic acid. The shelf life of these solutions is not known at this time, but they have been used after being stored for 2 weeks. Both of these solutions should have their own containers. About 100-150 ml of each should be placed in it's respective containers. A D.C. power supply that is capable of 6 volts and 1 amp should be used. It should have two long leads, Hydrochloric acid fumes/vapor are very corrosive. The containers are two crystal growing dishes that will hold approximately 300 ml. If the sample is not mounted metal tongs are used to hold and make electrical contact with the sample. If the sample is mounted, then a sharpened carbon rod or similar can be used. The counter electrode that should be used is either palladium coated titanium mesh, carbon, or platinum. All experiments were done with a carbon electrode. The negative of the power supply should be connected to the counter electrode. The positive should be connected to the tongs or sharpened carbon rod. The voltage on the power supply should be set to 3 volts. If the etching or polishing goes too fast, the voltage should be turned down slightly.

If the sample is not mounted and a carbon counter electrode is being used,grab it with the tongs so that the longest distance of the sample is being held. Position the sample so the polished face is 45°with respect to down and also it is facing the counter electrode. Place the counter electrode so that it is diagonal in the dish that is holding the electro polishing solution. Turn the power supply on. Move the sample back and forth somewhat quickly, but not so quickly that the sample face does not have full liquid contact. Do this for a ten count. Remove from the polishing solution and rinse with 18MΩ water. After rinsing it should be immediately returned to the Methanol.

If the sample is mounted and a carbon counter electrode is used, the sample should be placed at the bottom of the polishing solution face up. The sharpened carbon rod should make contact in the corner of the sample. Turn the power supply on and move the counter electrode around in the container to get an even polishing. Do this for a twelve count. Remove from solution, rinse with 18MΩ water and immerse immediately in Methanol.

It is possible to get a polish and etch from the electropolishing solution. The sample should be left in for a longer time. This was found at the end of the metallographic project and had a good result, but it was not followed up since it was the last sample.

8/13/93

## Etching

To etch the sample, use the same technique as was followed above, but in the etching solution. The time that the sample is left in the solution, 190 ml of 36% HCL and 10g Oxalic acid, should be shortened to at most 6 seconds. This process happens very quickly and etch times of 2 seconds have been too much at times. Staining of the sample will happen readily. If a slower etch is desired lower the voltage slightly. When the voltage is lowered too much more staining will occur. The viewable sections of the etch will be most likely at the corners, edges or at the edge of the stains. When done, rinse the sample thoroughly with 18MΩ water. Rinse off with acetone and dry.

## Other Precautions For Metallography

To minimize the amount of cross contamination, hands should be washed very thoroughly between each step after the 600 grit step.

The cloths and films need to be put on so they don't have any bubbles under them. Bubbles shorten the life of the cloth or film. The plate that the cloths are to be placed should be cleaned off and dried. The plates that will hold the films should be extremely clean. It has been noticed that pieces of cotton from a swab will cause a bubble. Once dirt is attached to the film it can not be removed. With clean hands place a small portion of the cloth or film on the plate and move your finger back and forth across it with some pressure. Move forward in this manner. If any discolorations occur it will most likely be a bubble. Pull back the film to release the bubble and continue.

When done all of the plates should be stored in ziploc type bags to avoid contamination of the cloths. The cloths will remain moist in their bags for some time, with maybe the exception of the Microcloth. The Microcloth should thoroughly soaked with 18MΩ water to remove most of the Mastermet and then tipped to allow most of the water off the cloth. In this way cloths can be used for some time.

It has been seen that the speed at which the sample is etched or electropolished can be determined by the direction that the sample is facing. The sample will etch slowest if it is facing up and fastest if pointing down. The closer the sample is to the counter electrode the faster the etching will go.

If a sample has been previously etched, it can be repolished starting with the rough polishing stage.

[signature] 8/13/93



8/13/93

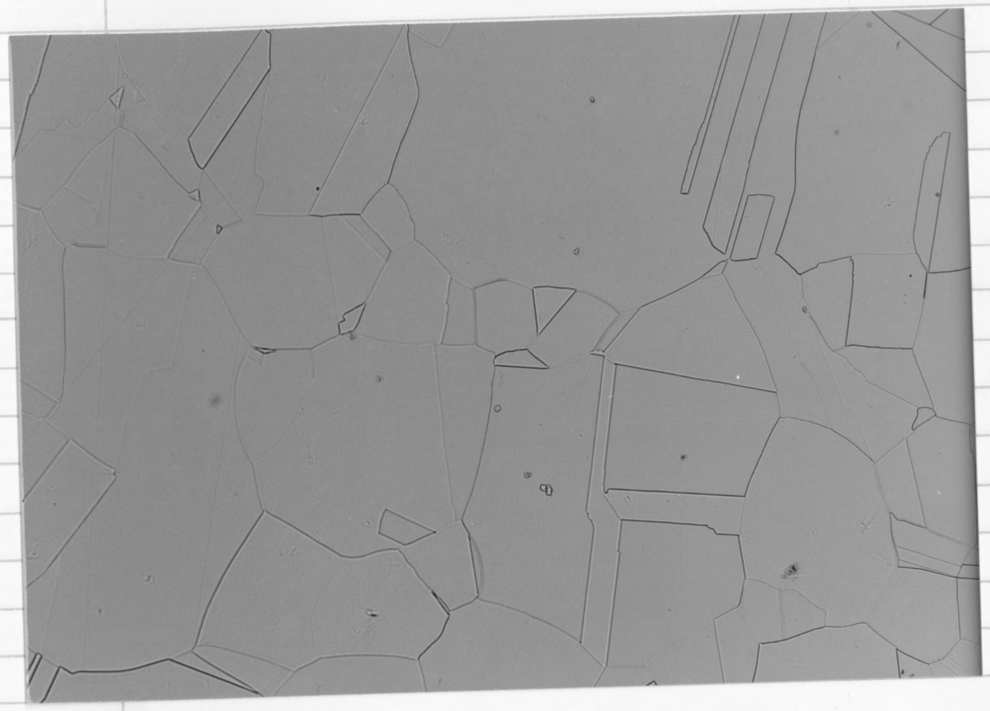
## List of Photographs for Alloy 825
Roll 97773
7/93

| | | | |
|---|---|---|---|
| 97773-1 | 750°~~C~~F/15hr | X100 | |
| 97773-2 | 750°~~C~~F/15hr | X100 | |
| 97773-3 | 750°~~C~~F/15hr | X50 | |
| 97773-4 | 750°~~C~~F/15hr | X50 | |
| 97773-5 | 750°~~C~~F/15hr | X200 | |
| 97773-6 | 750°~~C~~F/15hr | X200 | |
| 97773-7 | 750°~~C~~F/15hr | X400 | |
| 97773-8 | 750°~~C~~F/15hr | X400 | |
| 97773-9 | Mill Annealed | X200 | fine etch |
| 97773-10 | Mill Annealed | X400 | fine etch |
| 97773-11 | Mill Annealed | X400 | fine etch |
| 97773-12 | Mill Annealed | X100 | deep etch |
| 97773-13 | Mill Annealed | X200 | deep etch |
| 97773-14 | Mill Annealed | X400 | deep etch |
| 97773-15 | Mill Annealed | X400 | deep etch |
| 97773-16 | 750°~~C~~F/200hr | X50 | |
| 97773-17 | 750°~~C~~F/200hr | X100 | |
| 97773-18 | 750°~~C~~F/200hr | X200 | |
| 97773-19 | 750°~~C~~F/200hr | X400 | |
| 97773-20 | Sol. Annealed | X400 | |
| 97773-21 | Sol. Annealed | X200 | |
| 97773-22 | Sol. Annealed | X100 | |
| 97773-23 | Sol. Annealed | X50 | |
| 97773-24 | Sol. Annealed | X100 | |

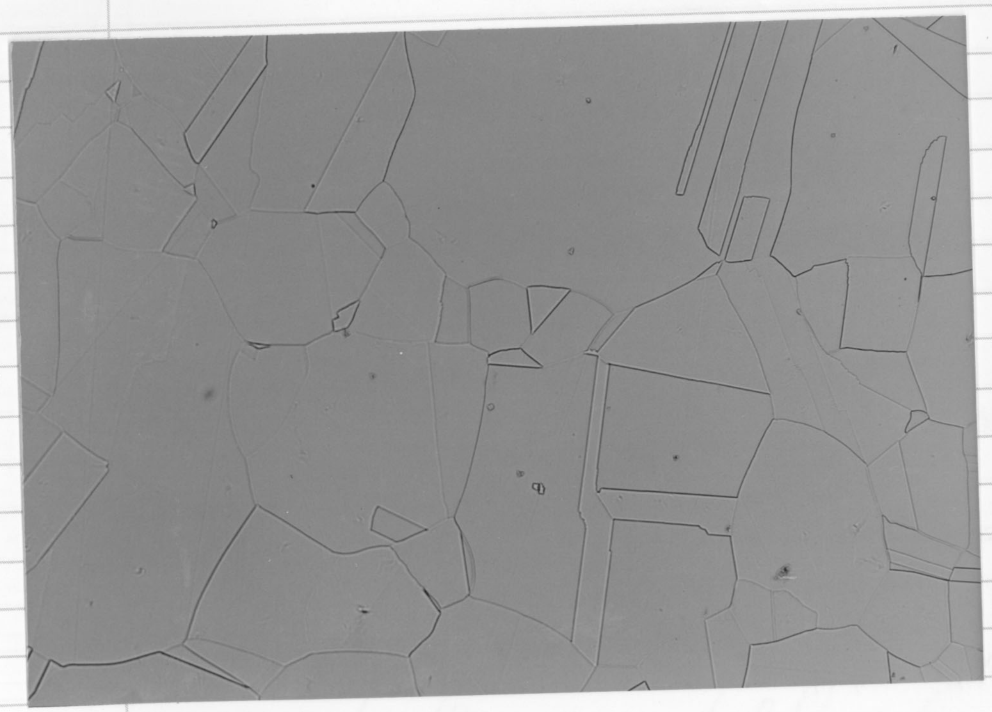
All samples that were heat treated after Solution Annealing were treated at 750°F not 750°C.

Metalographic Technique for 825, page 27 followed to obtain photographs.

[signature]
8/12/93



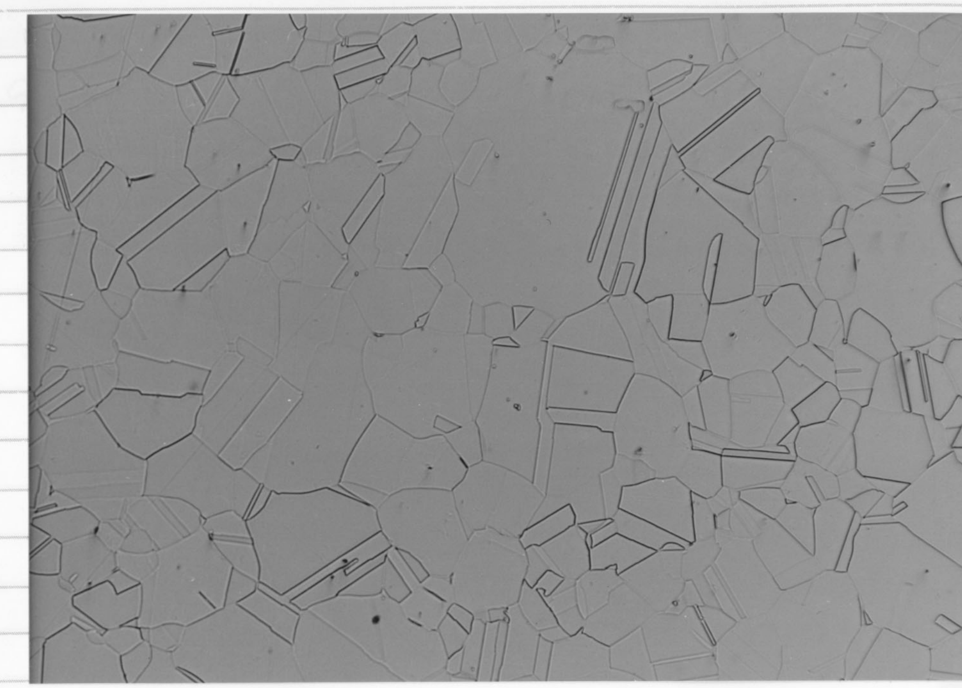
97773-1 750°F/15hrs X100



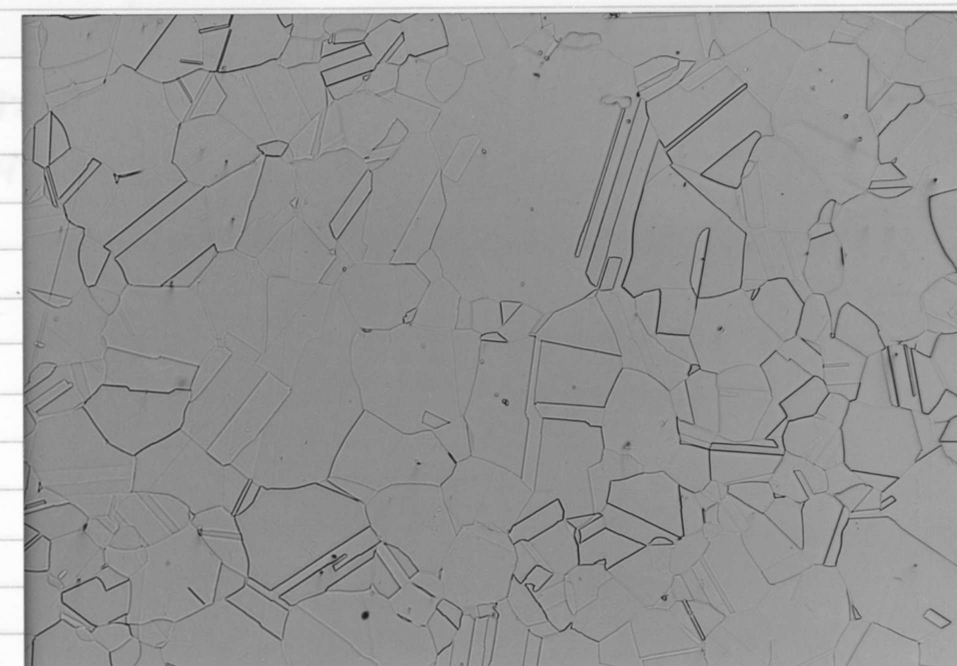
97773-2 750°F/15hrs X100

N. [illegible]
8/13/93



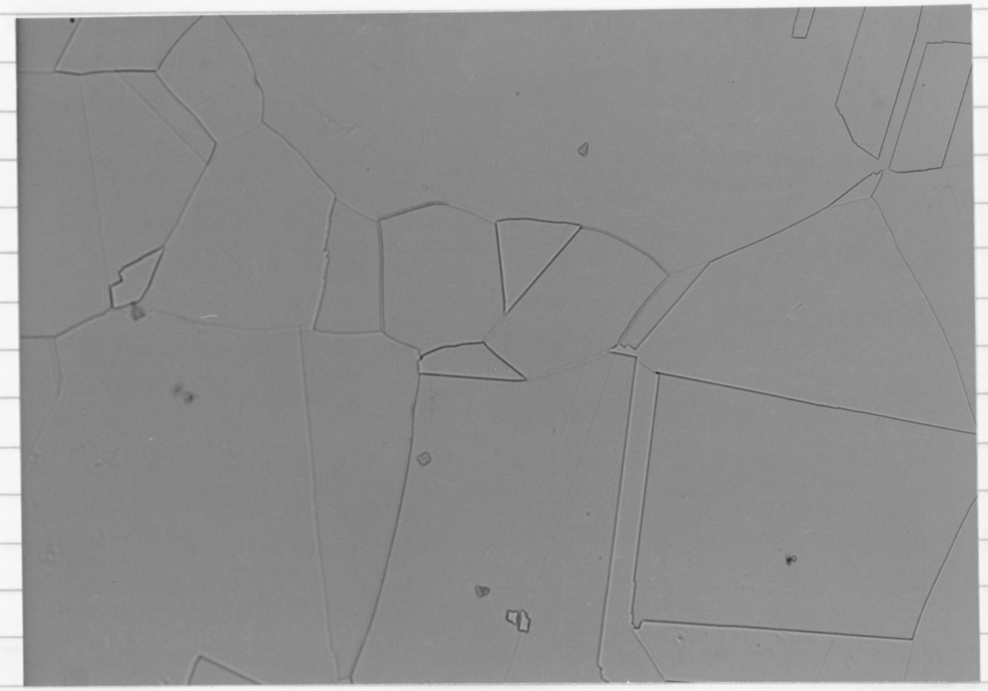


97773-3 750°F/15hrs X50

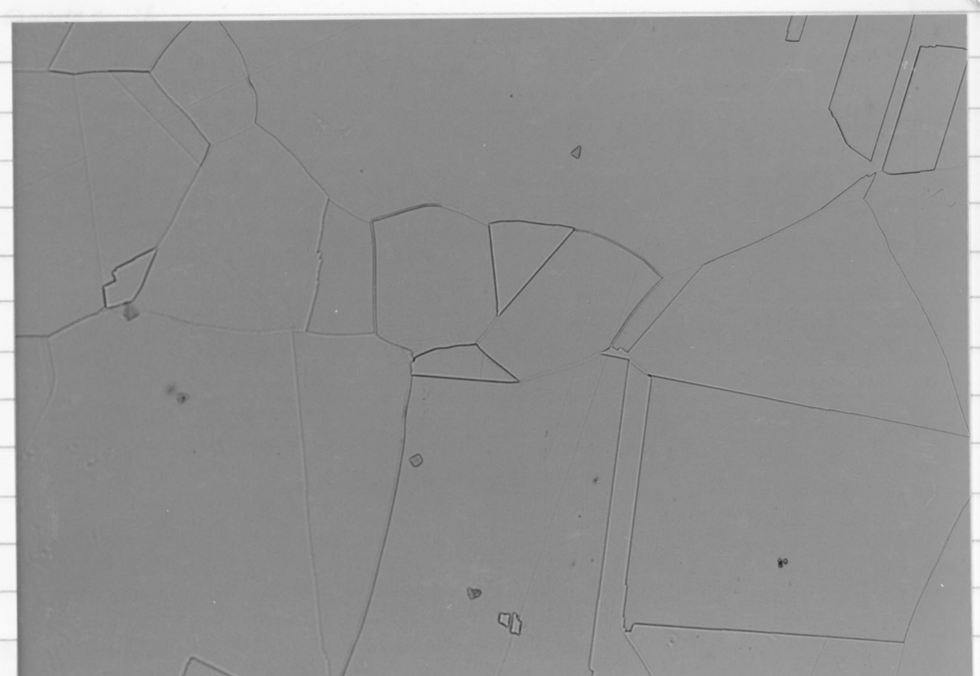


97773-4 750°F/15hrs X50

N. [illegible]
8/13/93



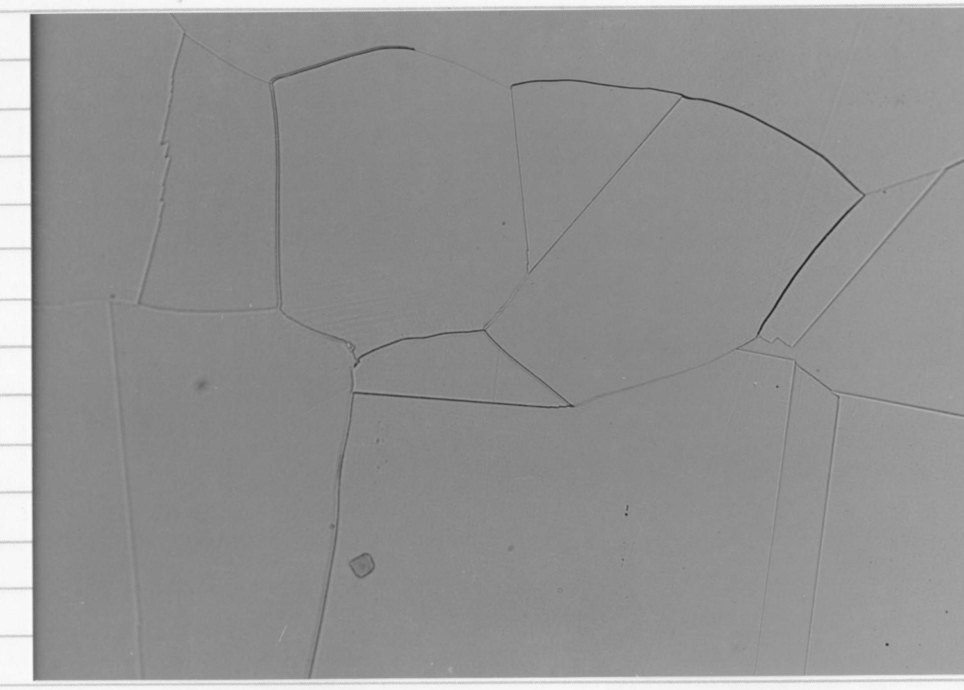
97773-5 750°F/15hrs X200



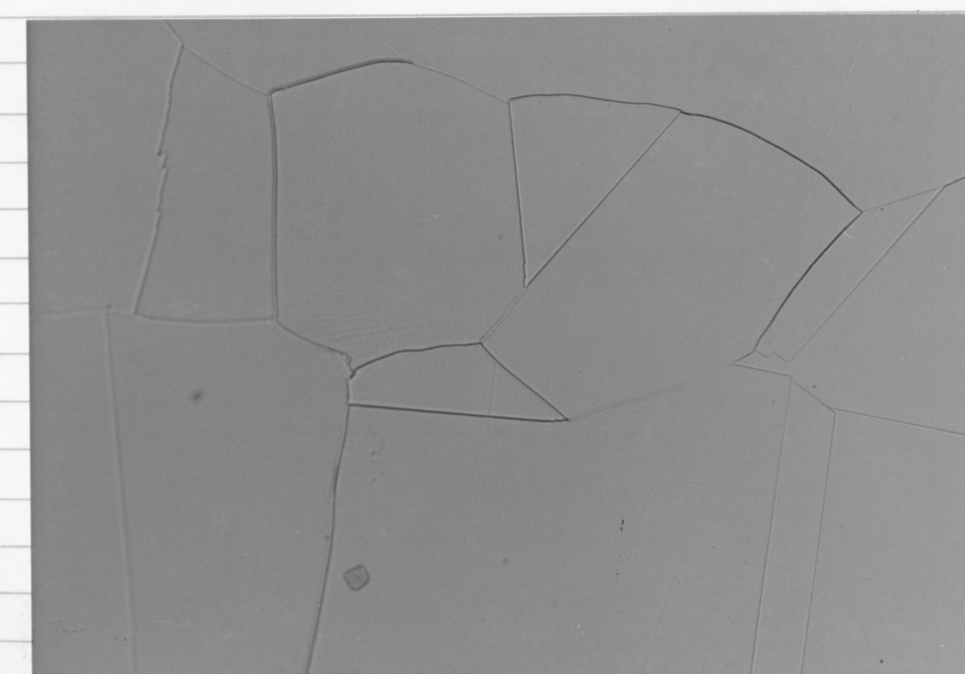
97773-6 750°F/15hrs X200

N. Sridhar
8/13/93



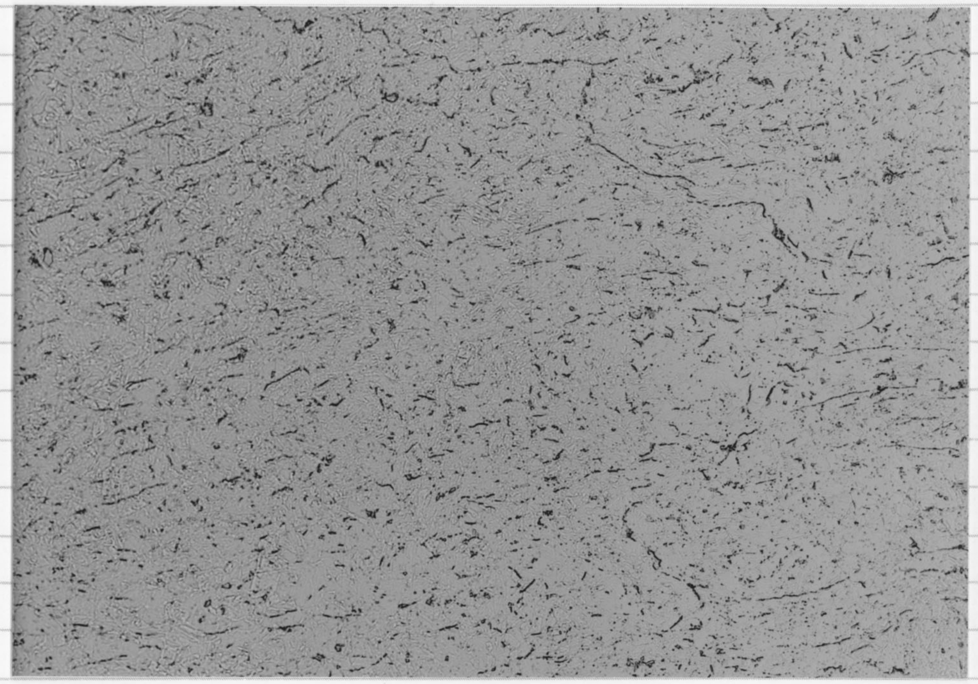


97773-7 750°F/15hrs X400

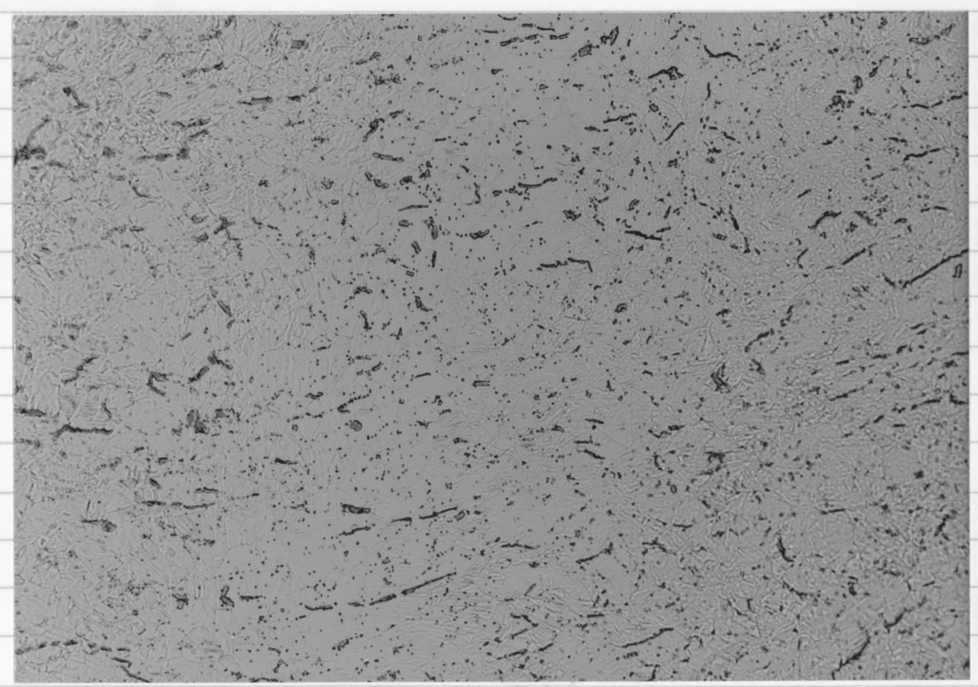


9777-8 750°F 15hrs X400

N. Sridhar
8/13/93



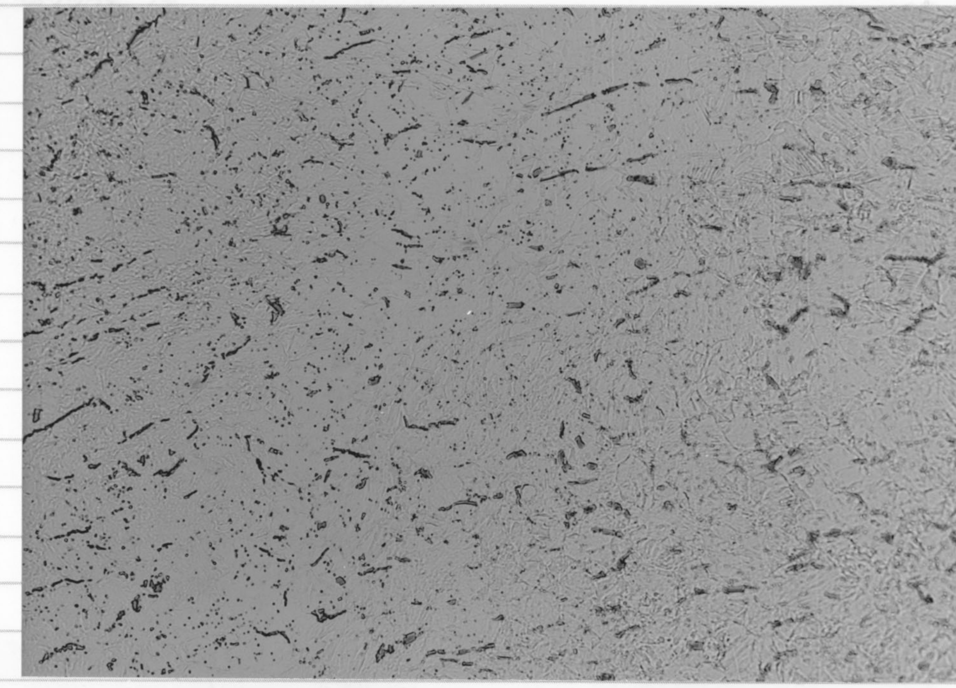
97773-9 Mill Annealed X200



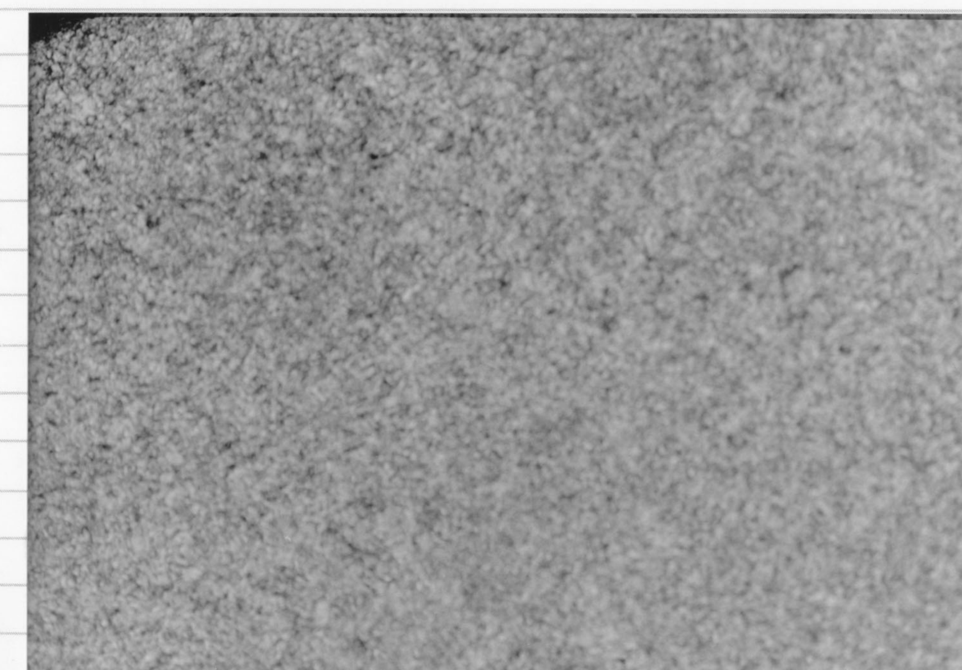
97773-10 Mill Annealed X400

N. [signature] 8/13/93



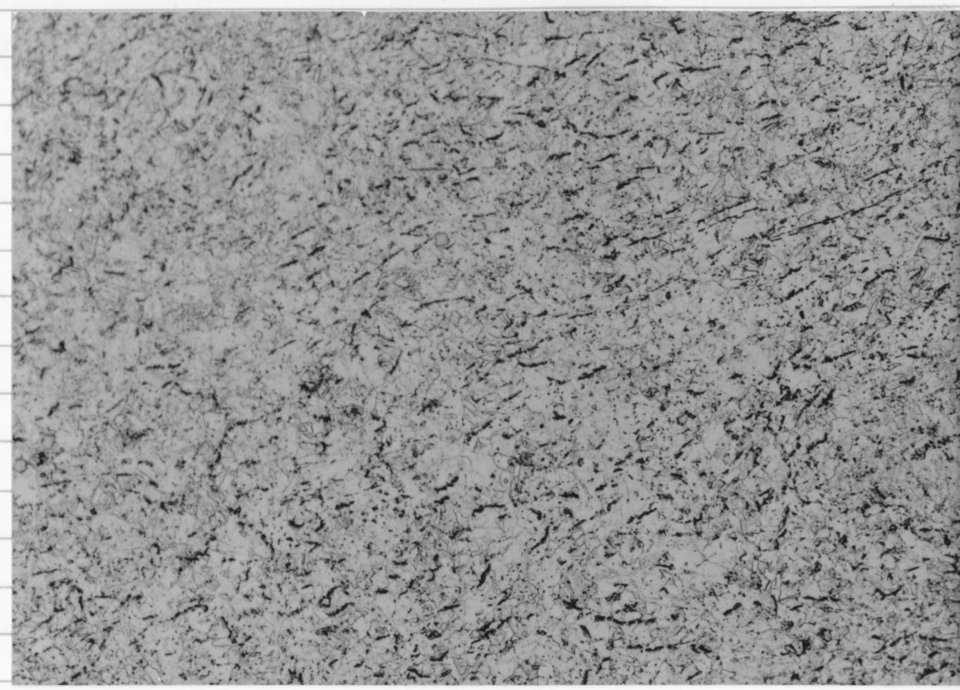


97773-11 Mill annealed X400

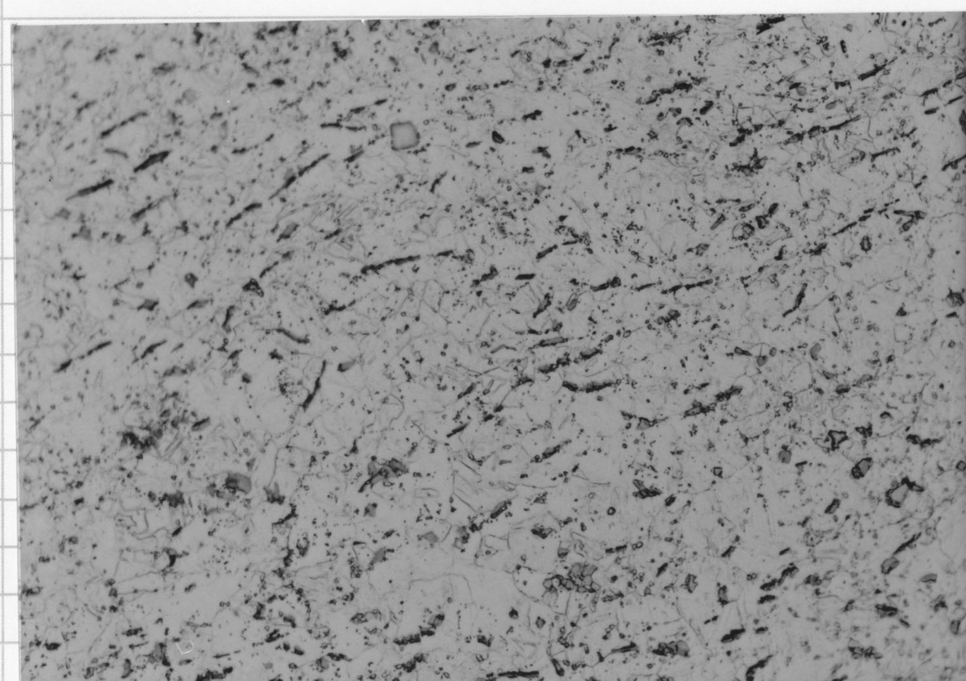


97773-12 Mill Annealed X 100

N. [signature] 8/13/93



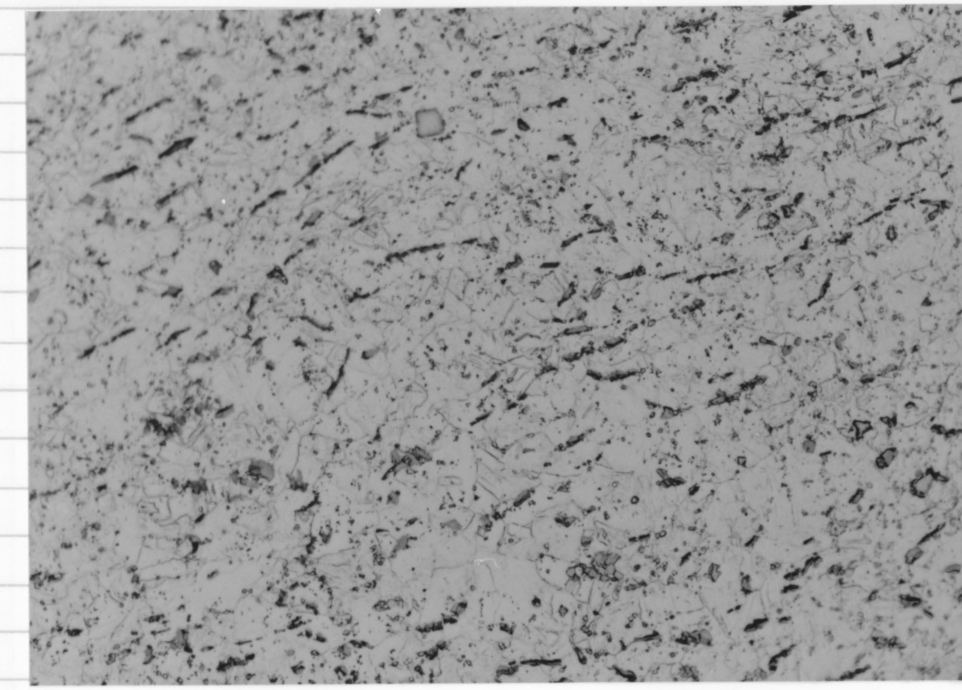
97773-13 Mill Annealed x200



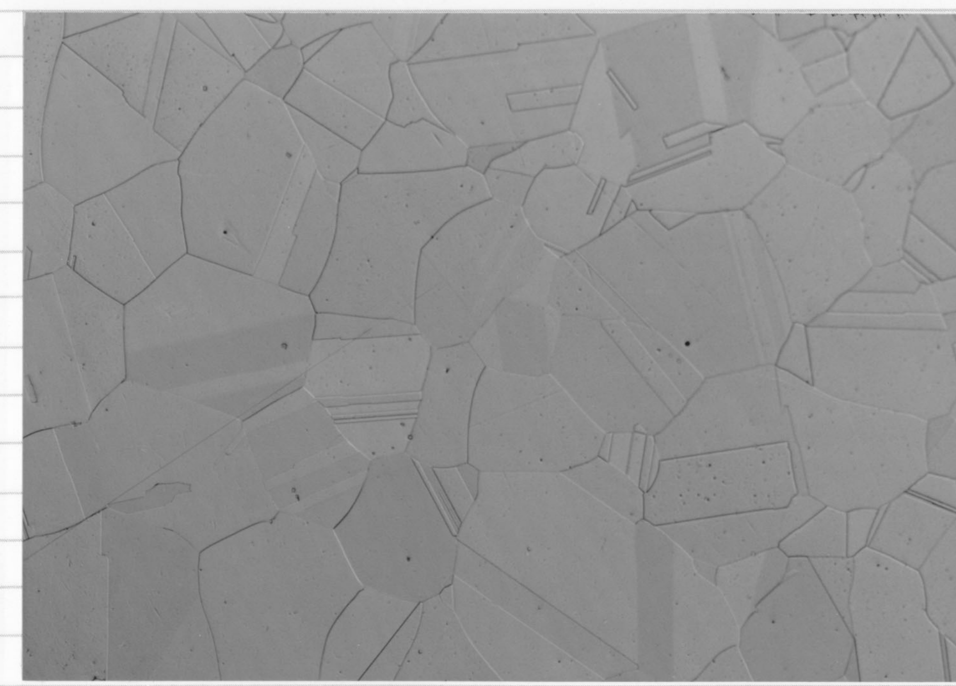
97773-14 Mill Annealed x400

N. Sridhar
8/13/93



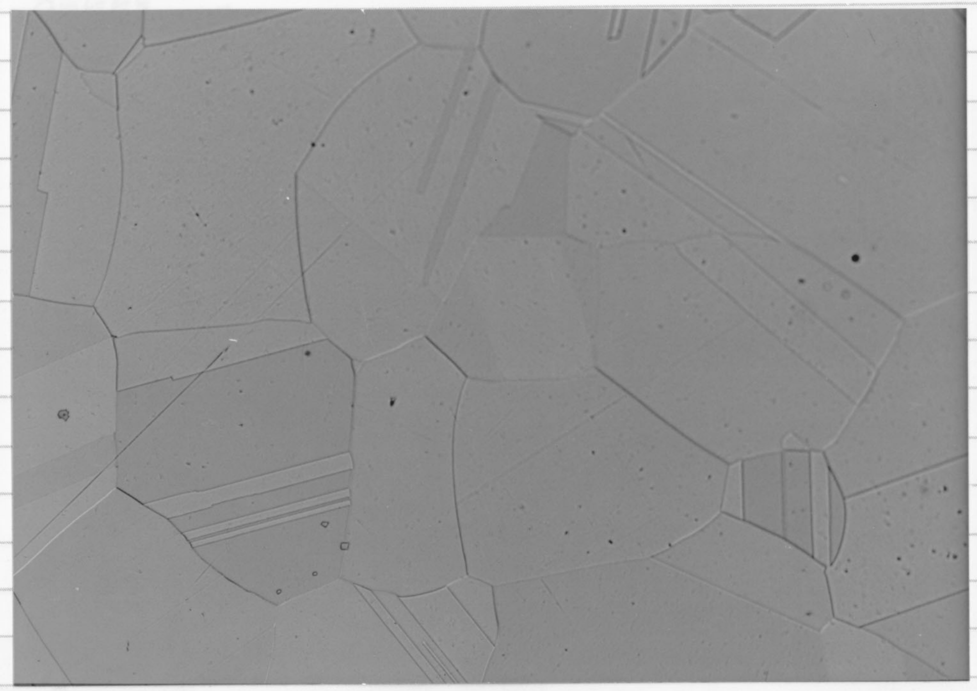


97773-15 Mill Annealed x400

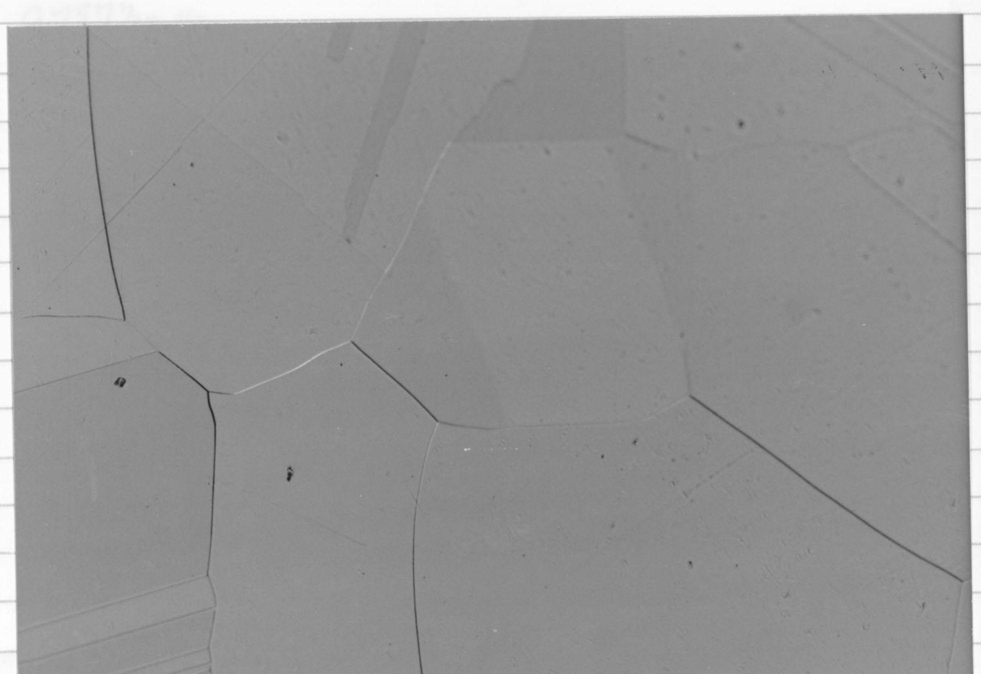


97773-16 750F/200hrs x50

N. Sridhar
8/13/93



97773-17 750°F/200hrs x100



97773-18 750°F/200hrs x 200

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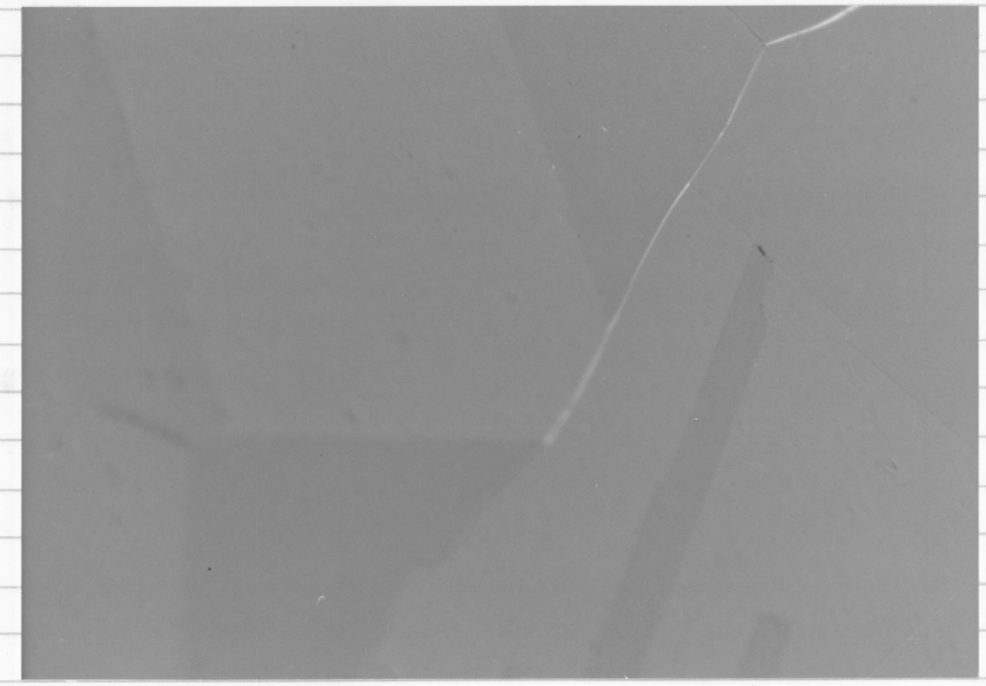
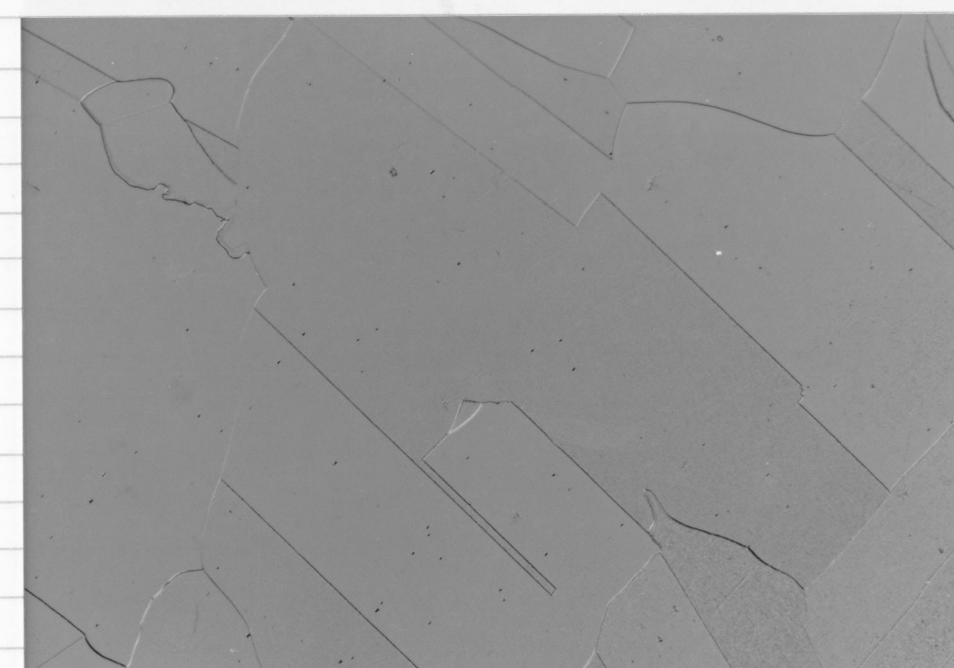
8/13/93





97773-19 750°F/200hrs x400



97773-20 Sol. Annealed x400

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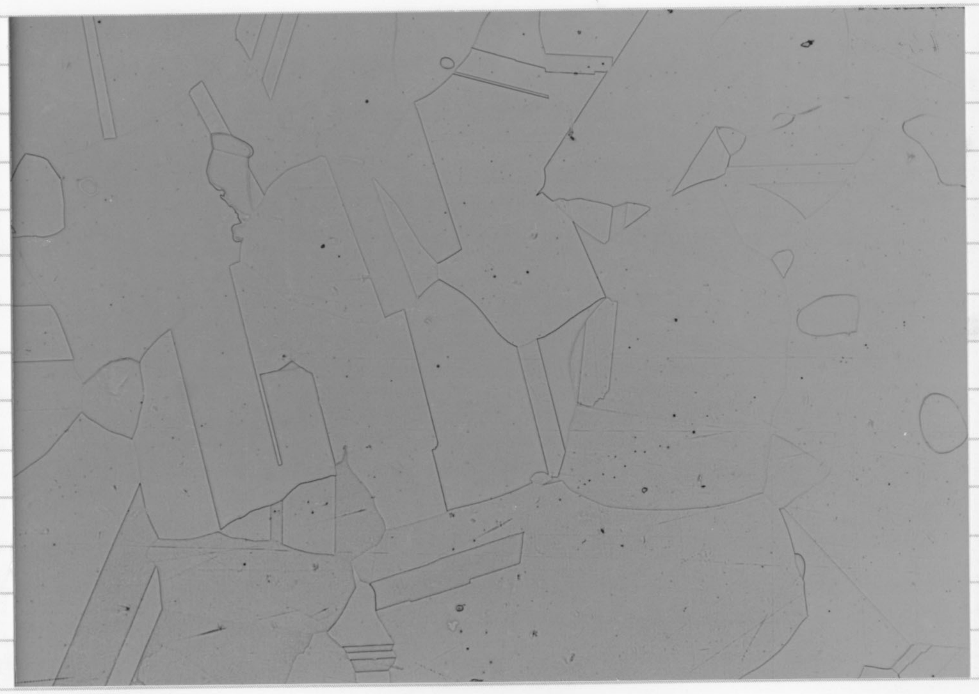
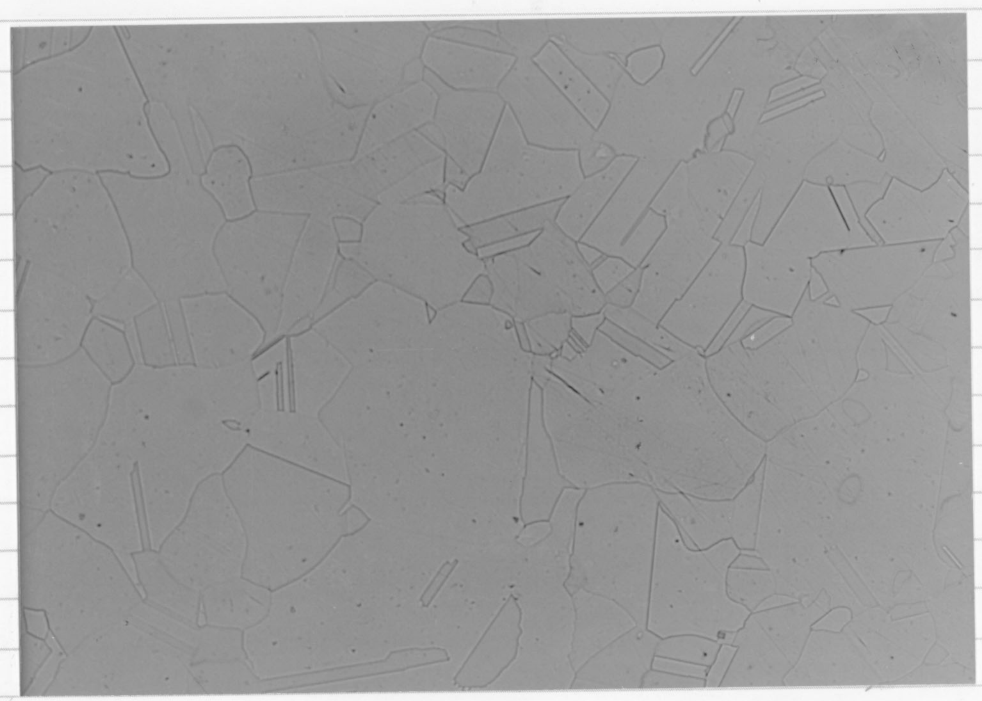
8/13/93



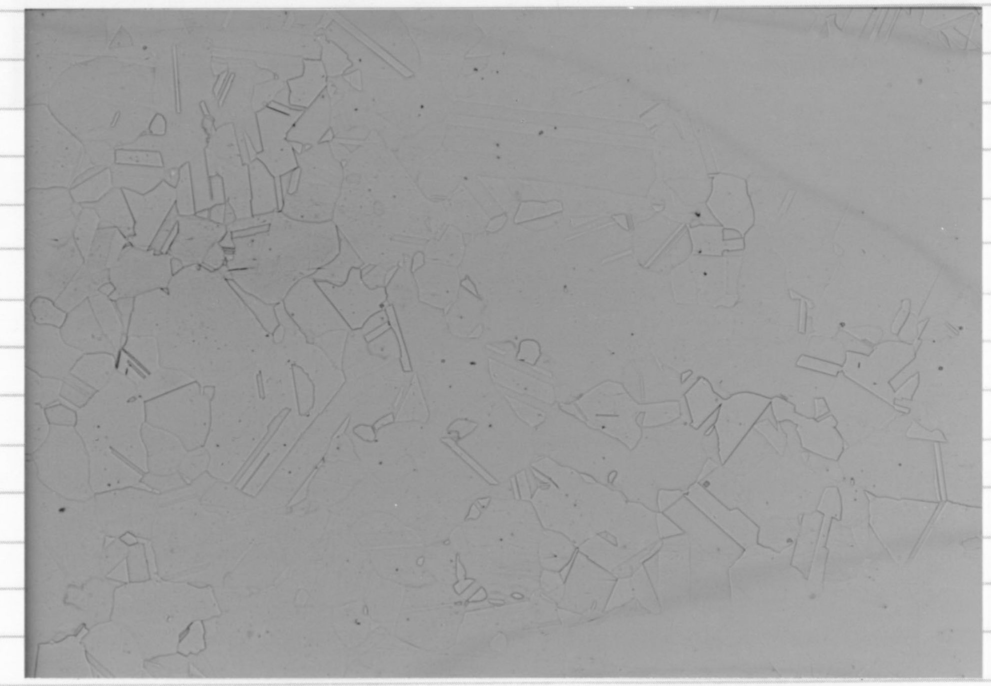
97773-21 Sol Annealed x 200



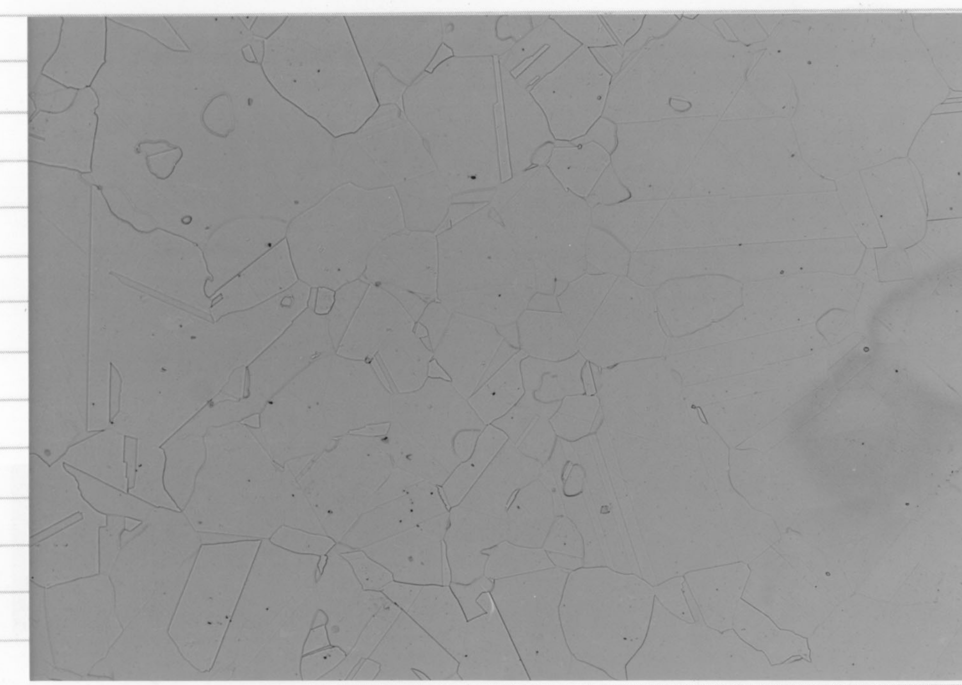
97773-22 Sol Annealed x 100

N. [signature] 8/13/93





97773-23 Sol Annealed X50



97773-24 Sol. Annealed X100

[signature] 8/13/93

8/13/93 List of Microstructures of Aged Alloy 825.

Heat Treated Alloy 825
7/93

| ID# | Sample | Mag. | Treated |
|---|---|---|---|
| 97844-1 | Mill Annealed | X200 | 1/93 |
| 97844-2 | Mill Annealed | X200 | 1/93 |
| 97844-3 | Mill Annealed | X400 | 1/93 |
| 97844-4 | Mill Annealed | X400 | 1/93 |
| 97844-5 | Mill Annealed | X100 | 1/93 |
| 97844-6 | Sol. Annealed, 15hr/750 | X200 | 1/93 |
| 97844-7 | Sol. Annealed, 15hr/750 | X200 | 1/93 |
| 97844-8 | Mill Annealed, 15hr/750 | X400 | 1/93 |
| 97844-9 | Mill Annealed, 15hr/750 | X200 | 1/93 |
| 97844-10 | Mill Annealed, 15hr/750 | X200 | 1/93 |
| 97844-11 | Sol. Annealed, 15hr/750 | X400 | 7/93 |
| 97844-12 | Sol. Annealed, 15hr/750 | X200 | 7/93 |
| 97844-13 | Sol. Annealed, 15hr/750 | X100 | 7/93 |
| 97844-14 | Sol. Annealed, 15hr/750 | X50 | 7/93 |
| 97844-15 | Sol. Annealed, 15hr/750 | X400 | 7/93 |
| 97844-16 | Sol. Anld, 15hr/750, 825CP9 | X200 | 7/93 |
| 97844-17 | Sol. Anld, 15hr/750, 825CP9 | X400 | 7/93 |
| 97844-18 | Sol. Anld, 15hr/750, 825CP9 | X100 | 7/93 |
| 97844-19 | Sol. Anld, 15hr/750, 825CP9 | X400 | 7/93 |
| 97844-20 | Sol. Anld, 15hr/750, 825CP9 | X200 | 7/93 |
| 97844-21 | Sol. Anld, 15hr/750, 825CP9 | X50 | 7/93 |
| 97844-22 | Sol. Anld, 15hr/750, 825CP9 | X50 | 7/93 |

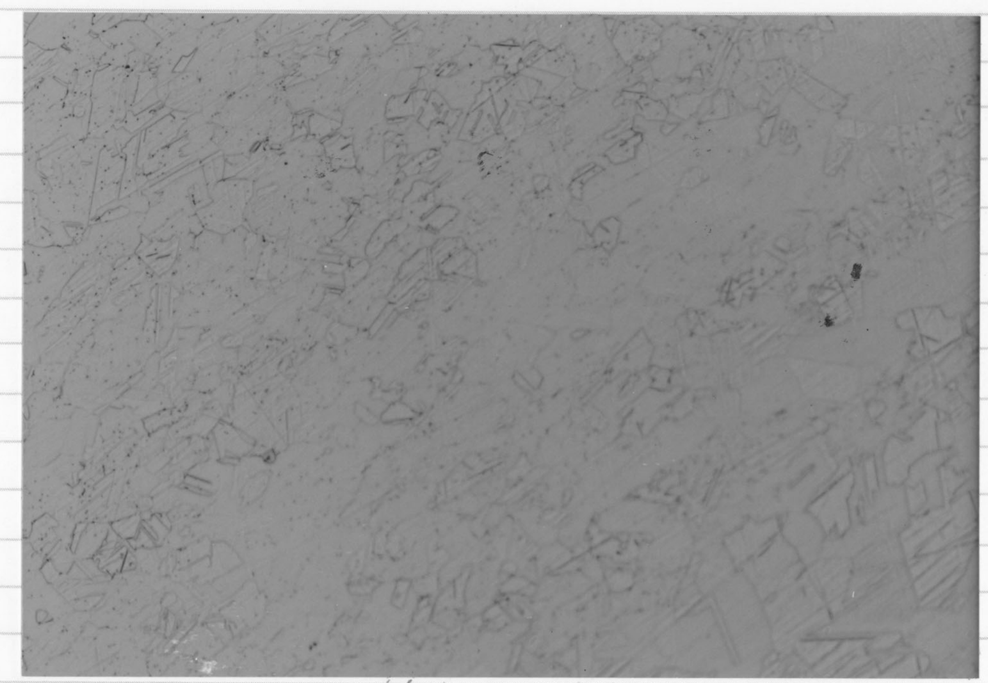
8/13/93

825CP9 is in Notebook 086

N. Sridhar
8/13/93

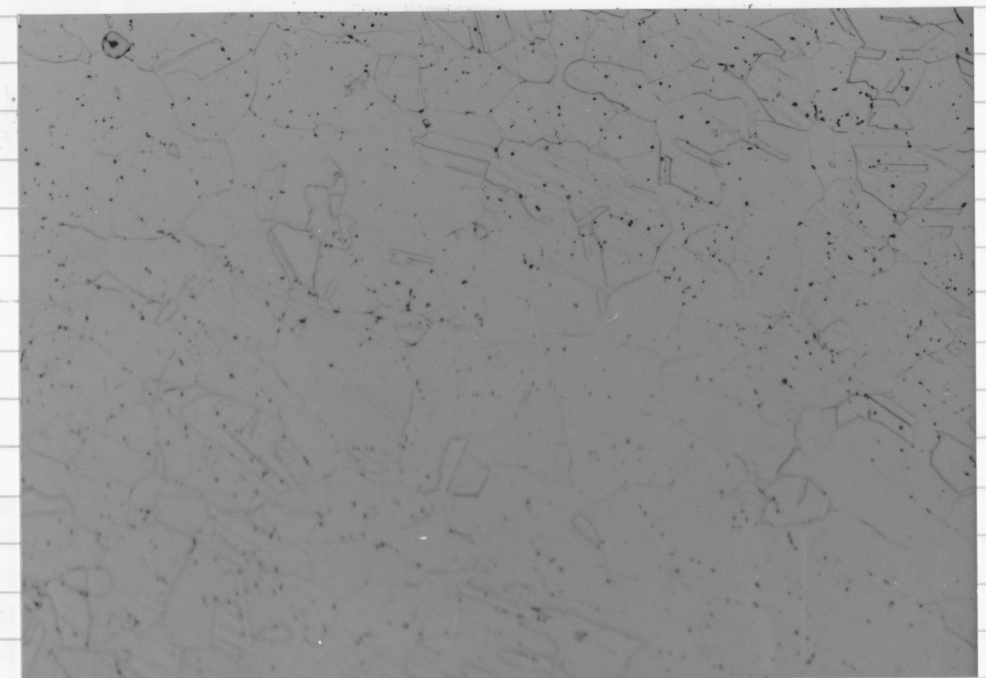


97844-1 Mill Annealed X 200

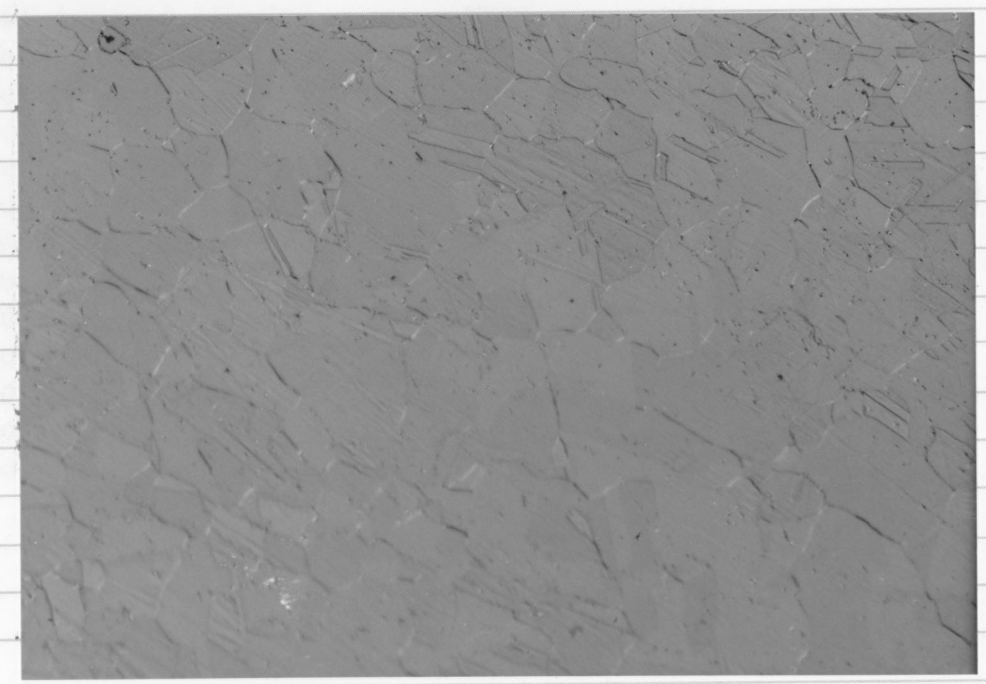


97844-2 Mill Annealed X 200

N. Sridhar
8/13/93



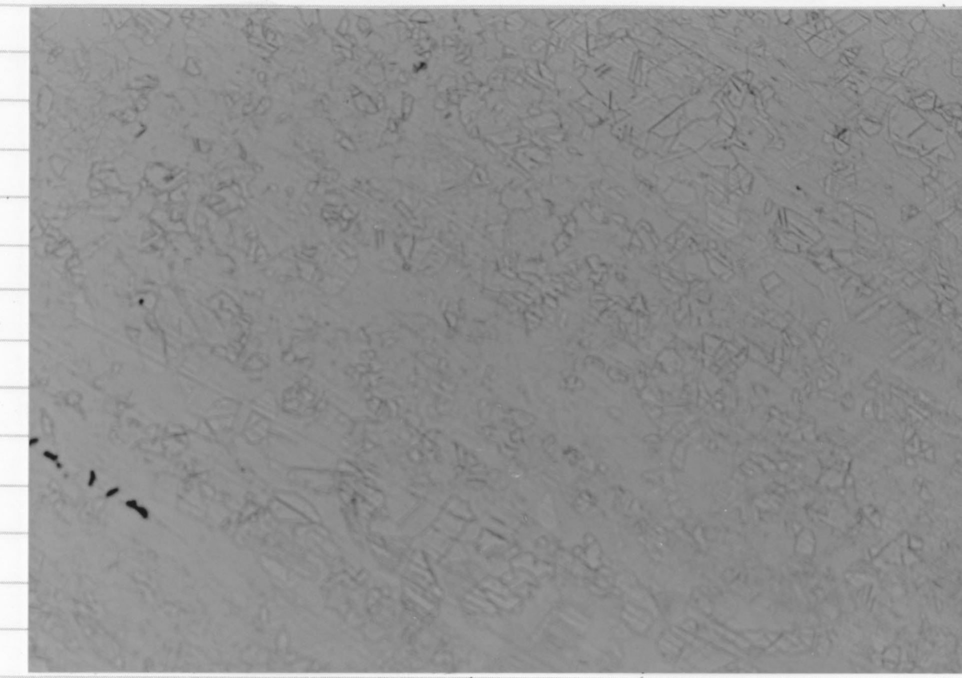
97844-3 Mill Annealed X400



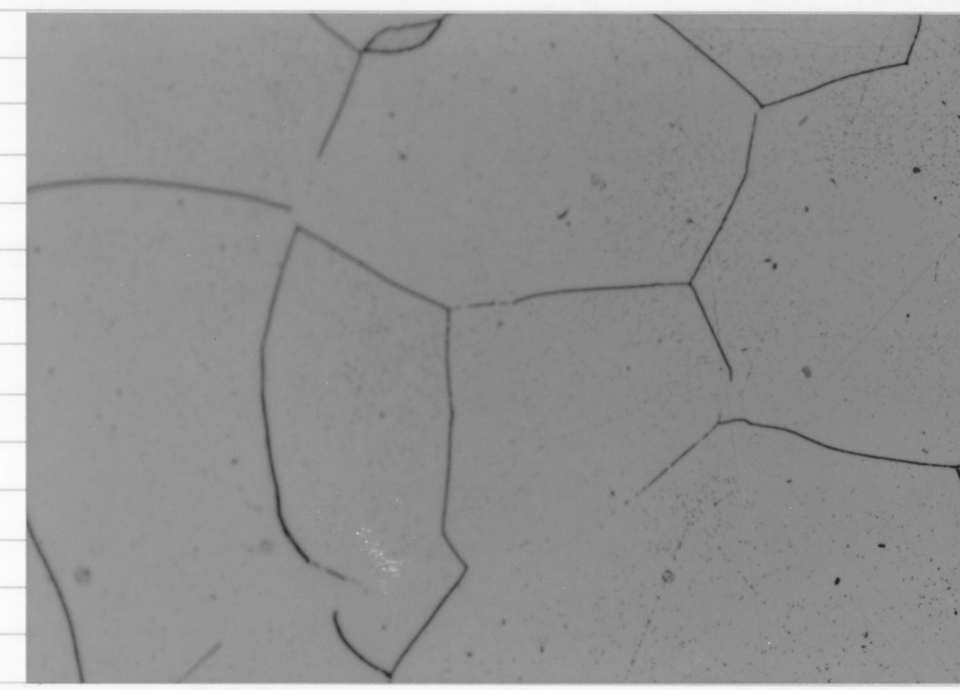
~~97844-3~~ Mill Annealed X400
97844-3

N. [illegible]
8/13/93



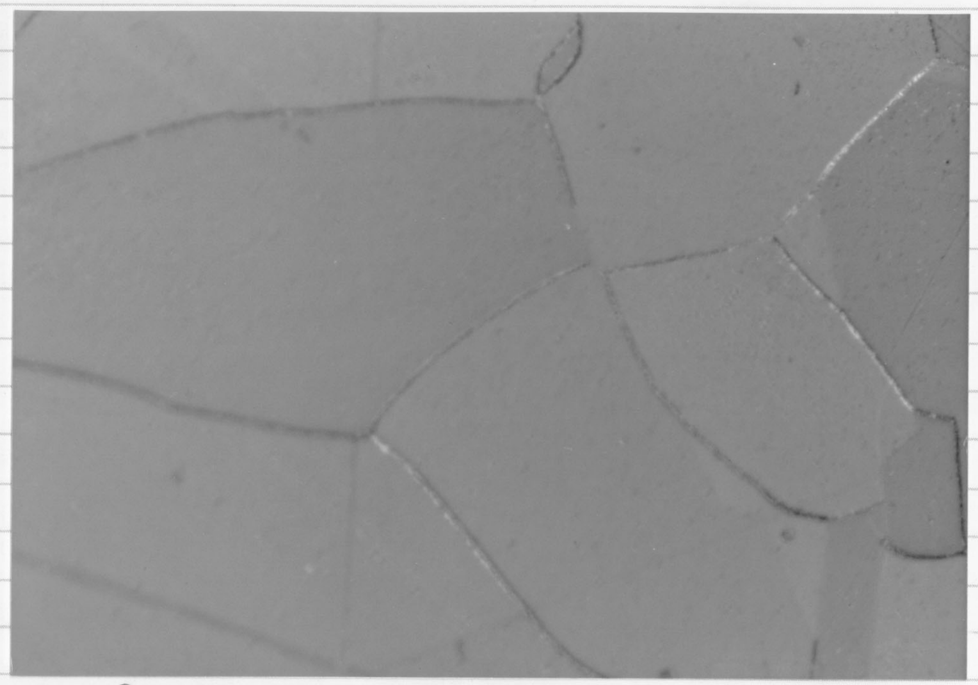


97844-5 Mill Annealed X100

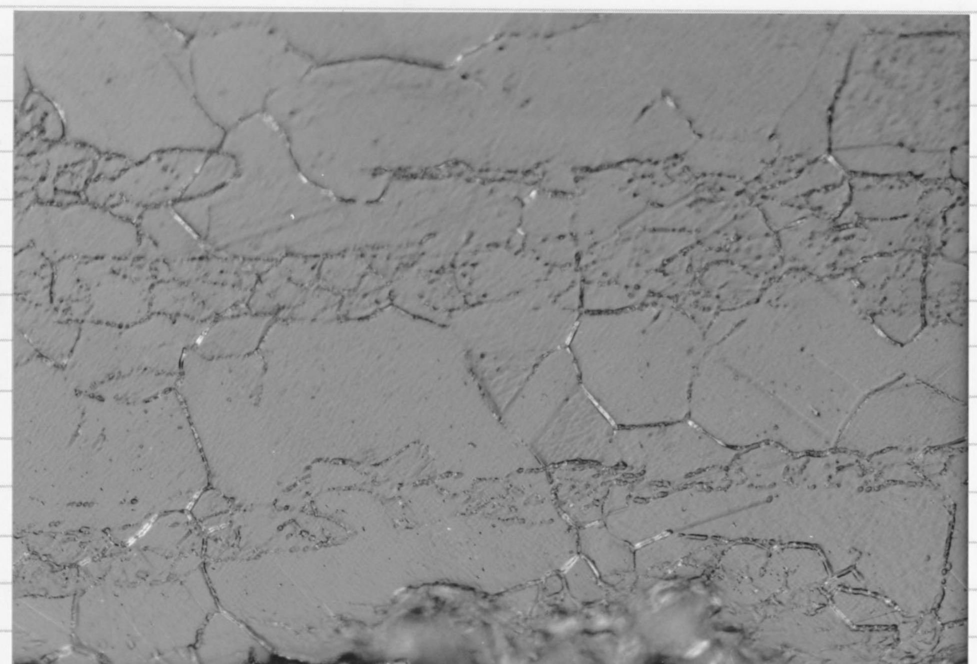


97844-6 SA → 750°C /15hrs X200

N. [illegible]
8/13/93



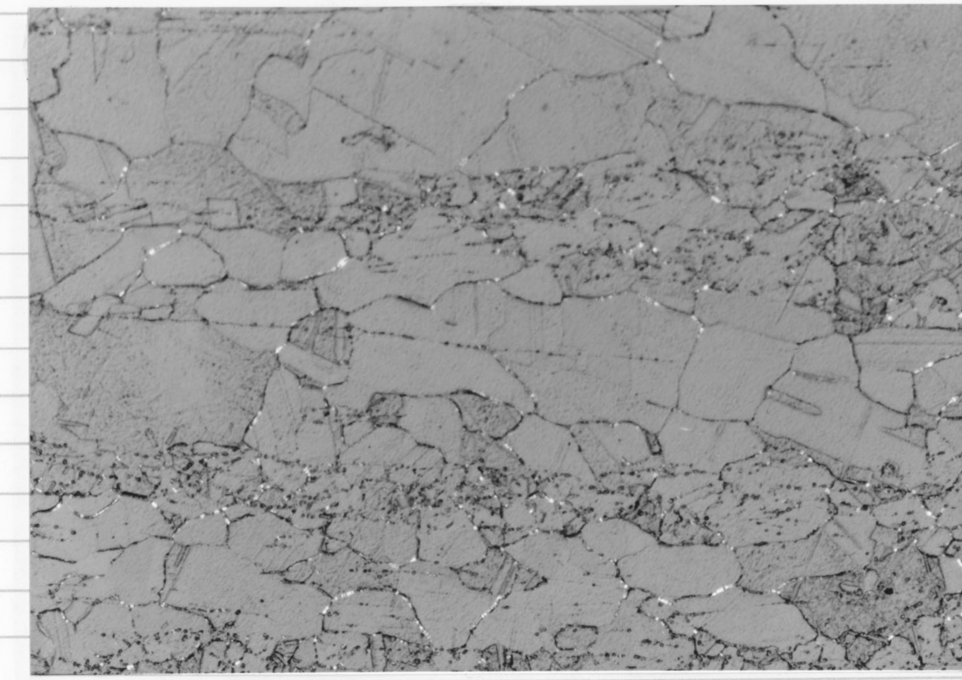
97844-7 SA → 750°C / 15hrs X200



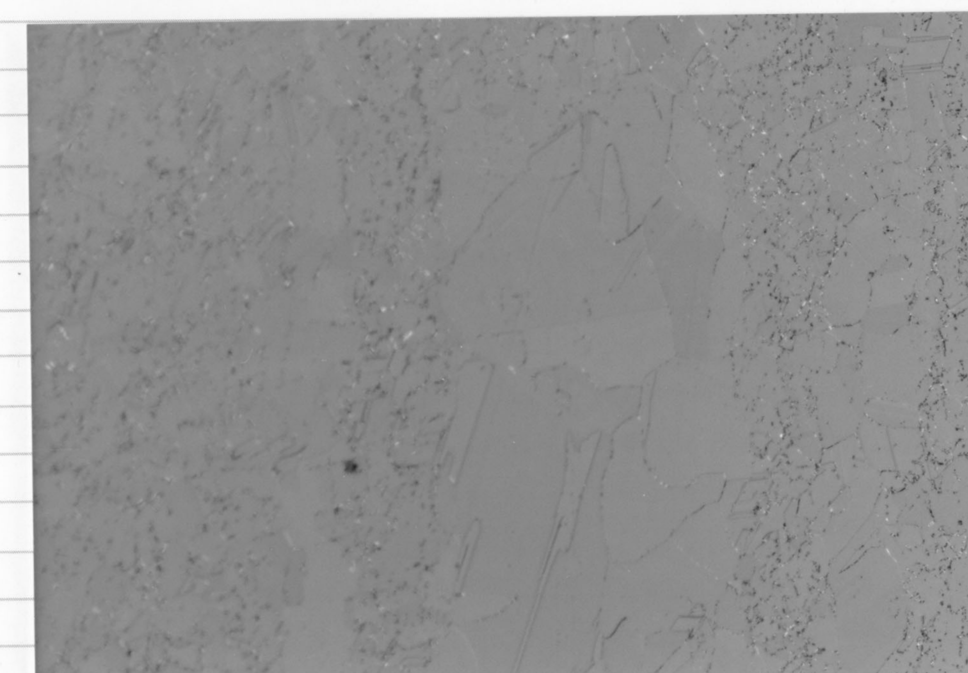
97844-8 MA → 750°C / 15hrs X 200

N. [signature]
8/13/93



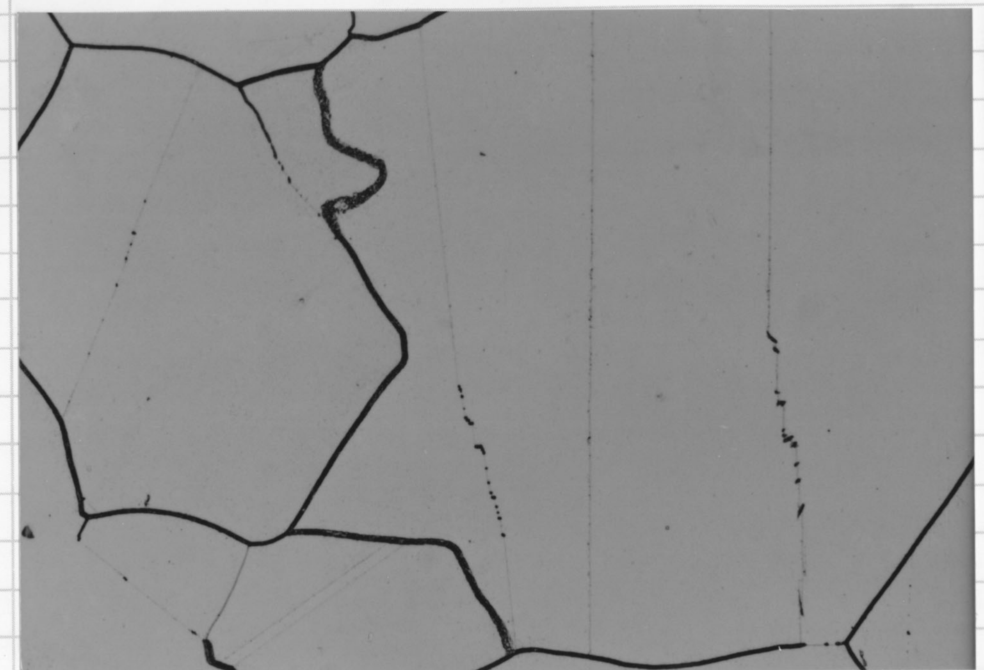


97844-9 MA → 750 / 15hrs ~~X 400~~ NK 8/13
X200

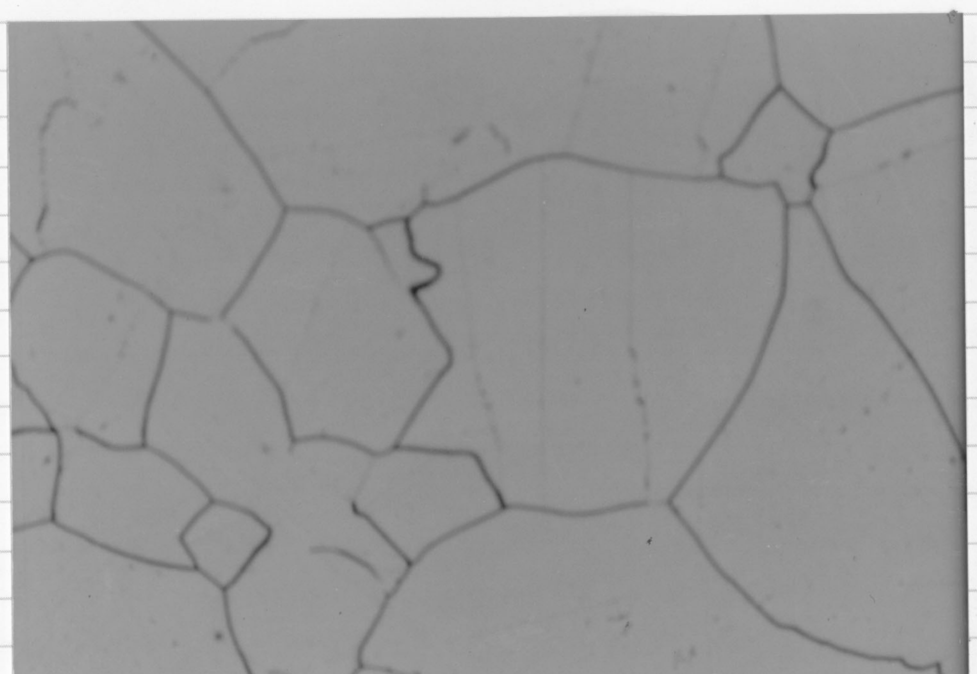


97844-10 MA - 750°C / 15hrs X 200

N. [signature]
8/13/93



~~97844-10~~ PK 8/13
97844-11 SA→ 750°C/15hrs X400
CNWRA#1



97844-12 SA→ 750°C /15hrs X 200
CNWRA#1

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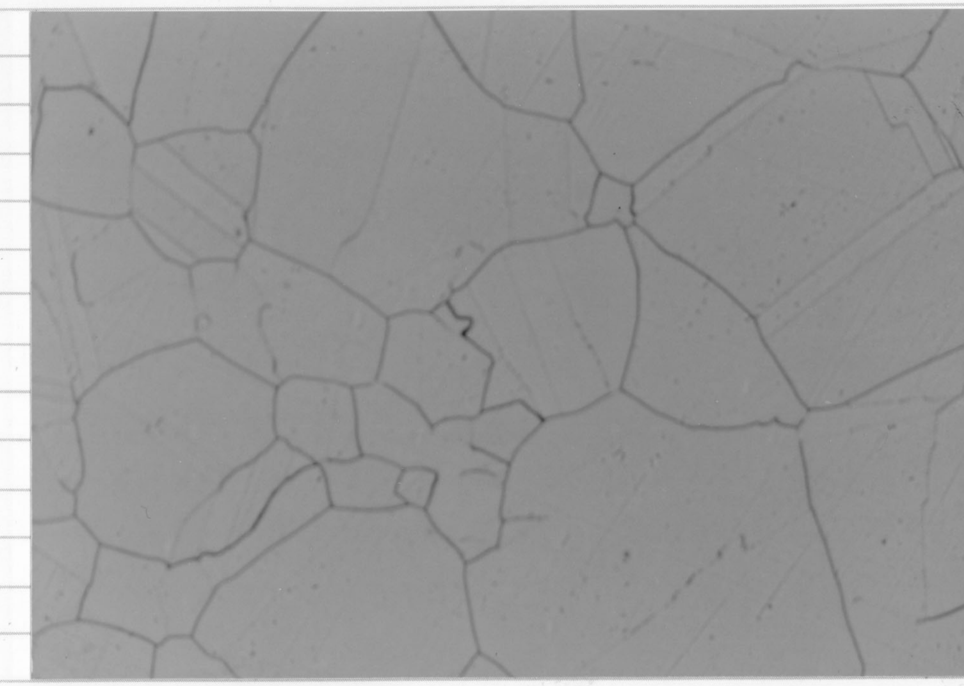
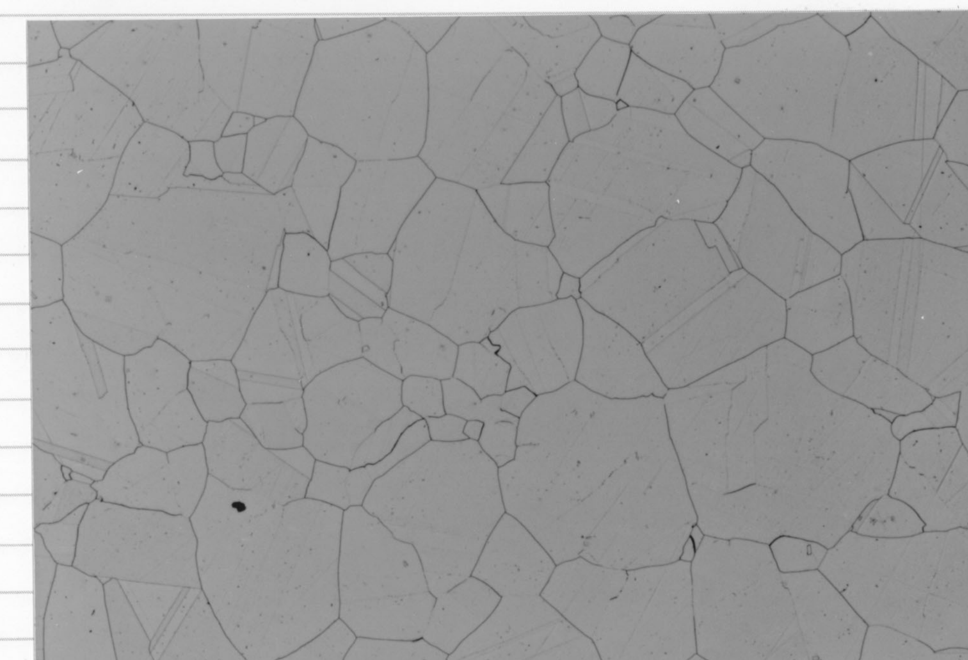
8/13/93





97844-13 SA→ 750°C /15hrs X100
CNWRA#1



97844-14 SA→ 750°C/15hrs X50
~~CNWRA#1~~ PK 8/13
CNWRA#1

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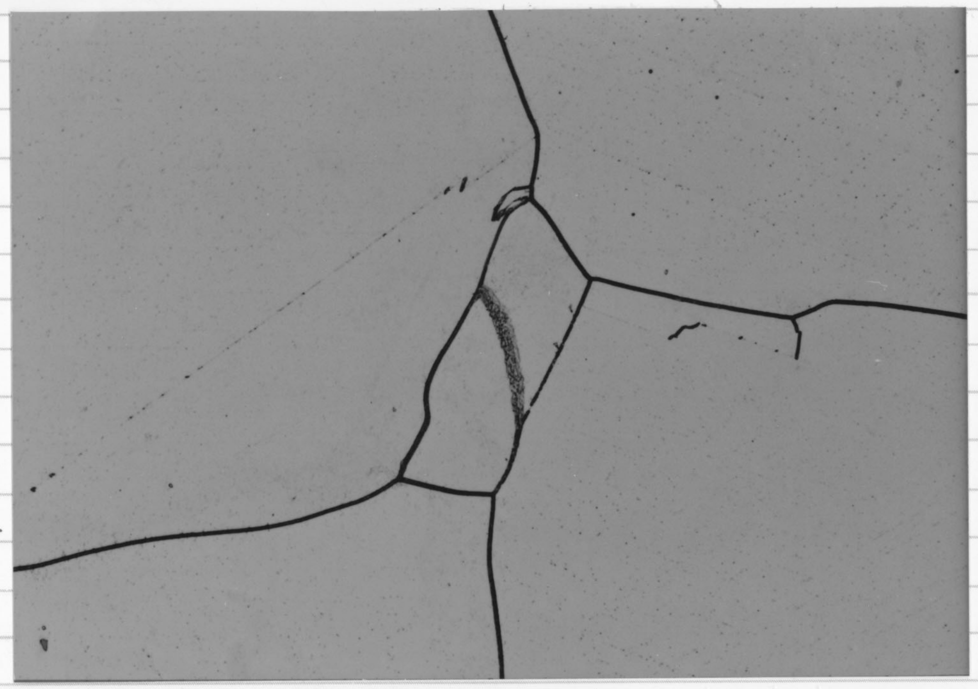
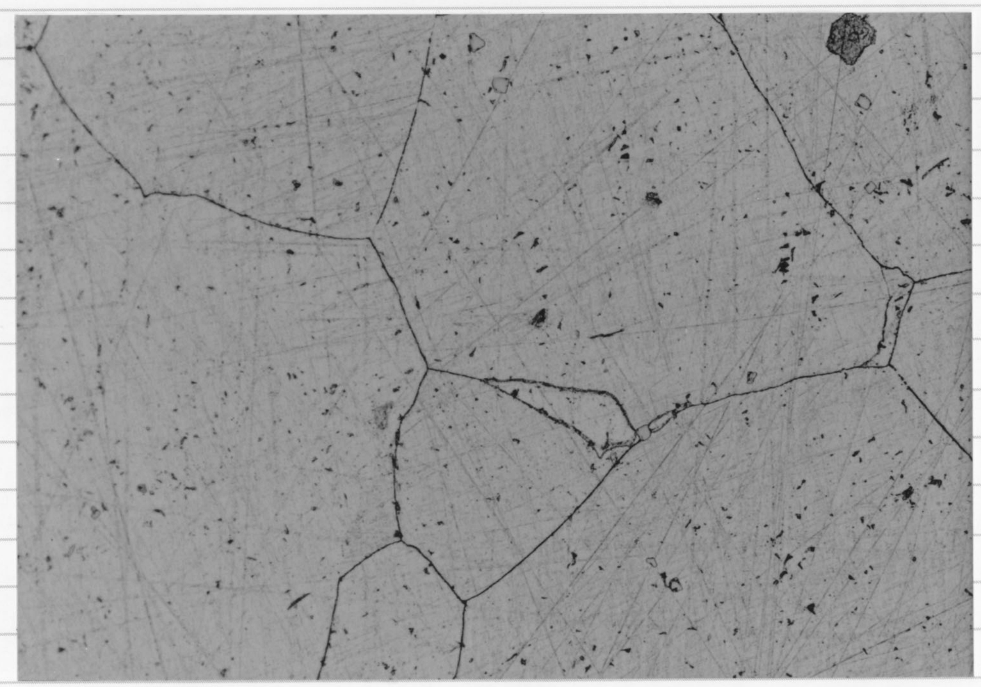


97844-15 SA→ 750°C/15hrs X400
CNWRA #1



97844-16 SA→ 750°C/15hrs X 200
CNWRAA 1 ~~(825CPA~~ → NK 8/13
(825 CP9)

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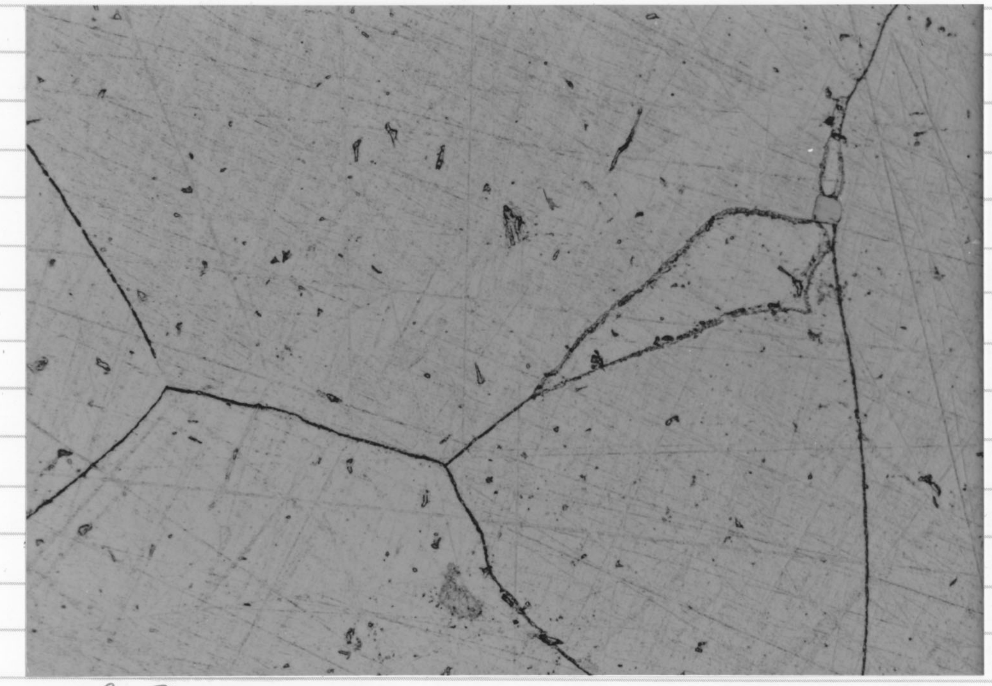
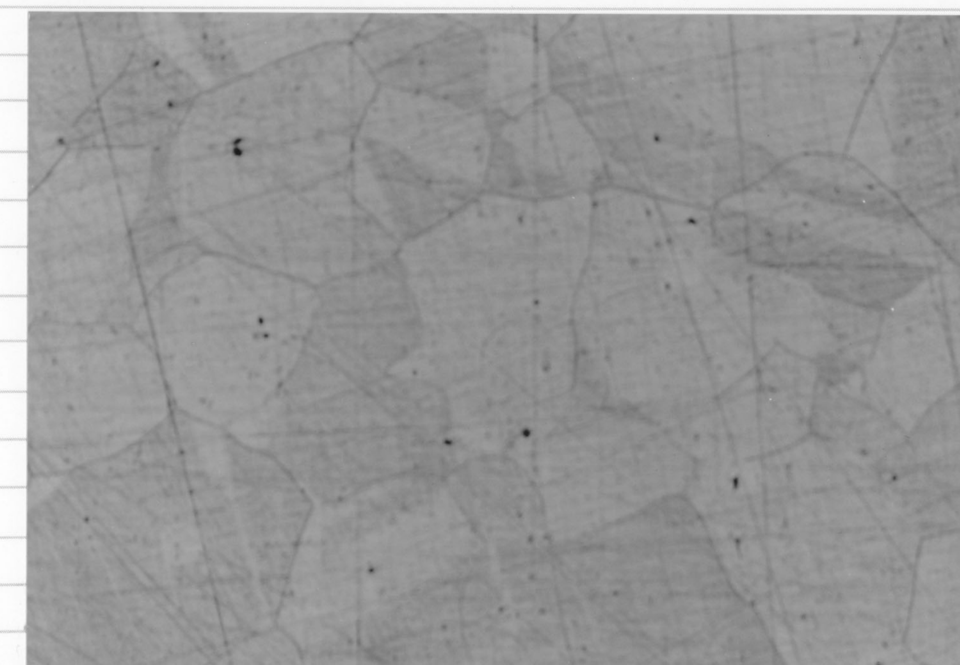
8/13/93





97844-17 SA→ 750°C/15hrs X 400
CNWRA#1 (825 CP9)



97844-18 SA→ 750°C/15hrs X 100
CNWRA#1 (825 CP9)

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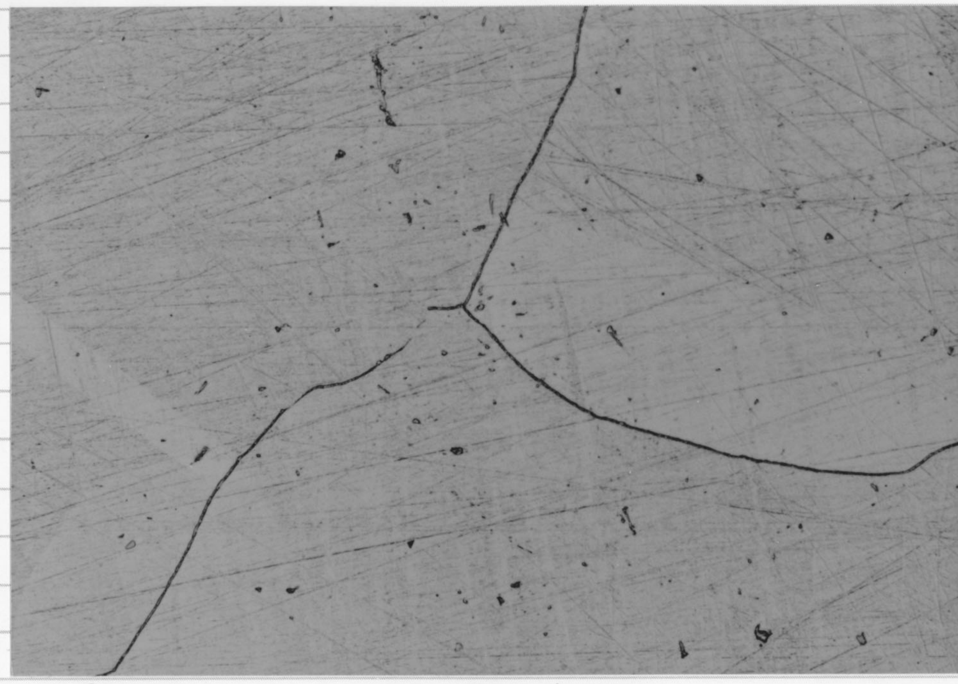
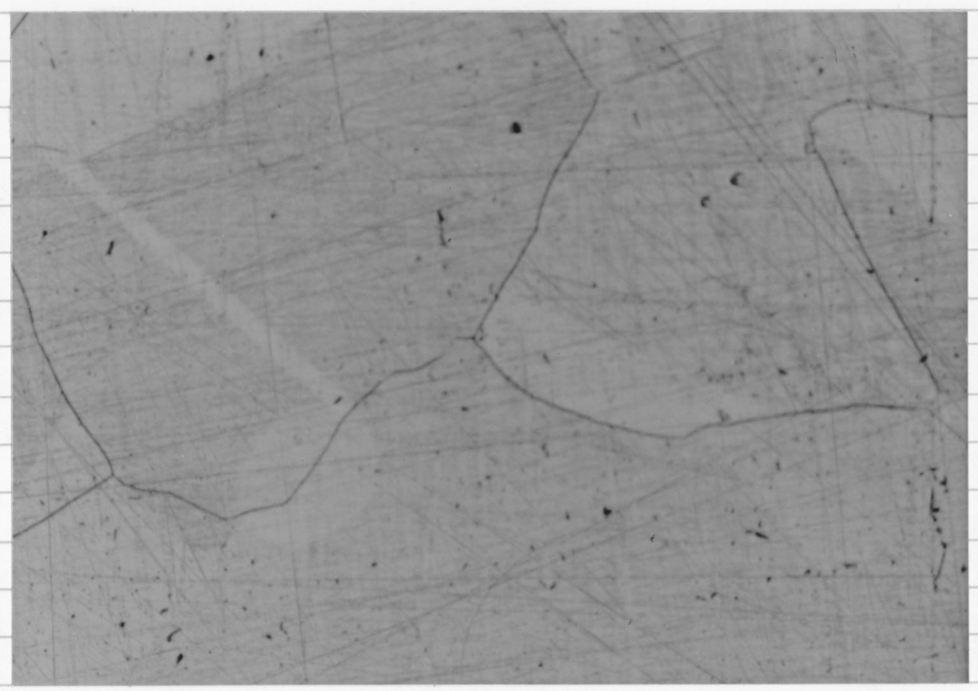
8/13/93



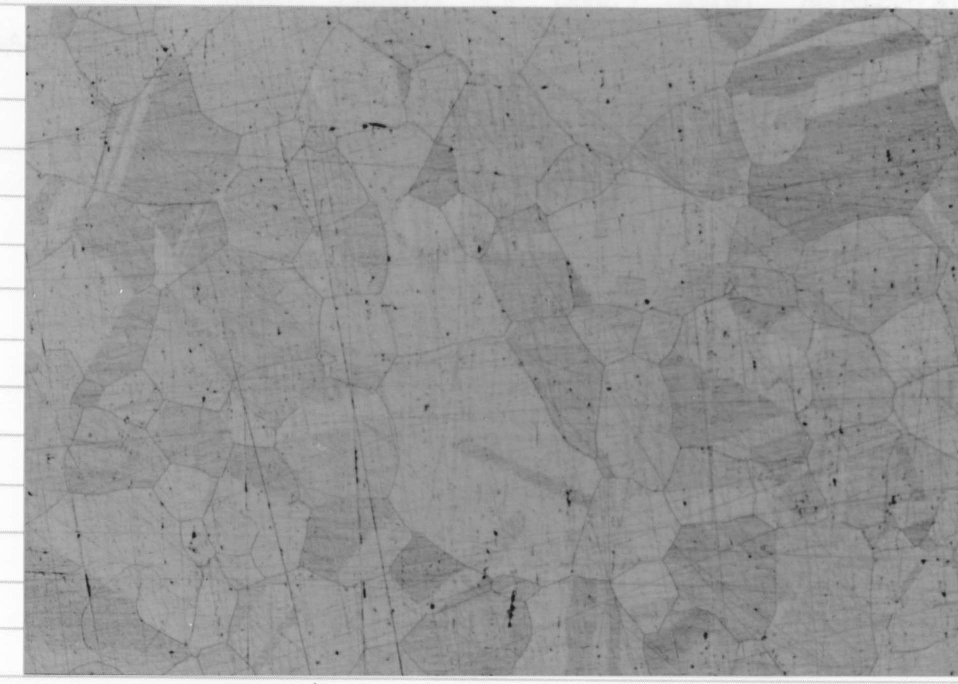
97844-19 SA → 750°C/15hrs X400
CNWRA# 1 (825CP9)



97844-20 SA → 750°C/15hrs X 200
CNWRA#1 (825CP9)

[signature]
8/13/93





~~97844 →~~ 8/13
97844-21 SA → 750°/15hrs X50
CNWRA#1 (825CP9)

97844-22 SA → 750°/15hrs X50
CNWRA#1 (825CP9)

[signature] 8/13/93

4/27/94 0.5 inch Thick Plates of Alloys Tested Thus Far in The IWPE Program. Vendor and Independant Lab Analyses.

CHEMICAL COMPOSITION IN WT% OF ALLOYS IN IWPE

| MATERIAL | 304L | 304L | 316L | 316L | IN825 | IN825 | HA C-22 | HA C-22 | CDA 102 | CDA 102 | CDA 613 | CDA 613 | CDA 715 | CDA 715 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ORIGIN | G.O.CARLSON | | EASTERN STAINLESS | | INCO/METAL GOODS | | HAYNES/CORR MATLS | | | | AMPCO METAL | | REVERE | |
| HEAT NO. | T0954 | | P80746 | | HH4371FG | | 2277-8-3175 | | | | M5459 | | 7037-61326 | |
| ELEMENTS | VENDOR | KAWIN | VENDOR | KAWIN | VENDOR | KAWIN | VENDOR | KAWIN | VENDOR | KAWIN | VENDOR | KAWIN | VENDOR | KAWIN |
| Ag | | | | | | | | | | | | <0.01 | | |
| Al | | <0.01 | | <0.01 | 0.07 | 0.05 | | 0.18 | | | 6.66 | 7.05 | | |
| As | | | | | | | | | | | | <0.01 | | <0.01 |
| B | | | | | | | | | | | | | | |
| C | 0.022 | 0.029 | 0.014 | 0.019 | 0.010 | 0.013 | 0.004 | 0.015* | | | | 0.005 | 0.013 | 0.019 |
| Cb | | | | | | | | | | | | | | |
| Cd | | | | | | | | | | | | <0.001 | | <0.001 |
| Co | | | 0.17 | | | | 0.09 | 0.87 | | | | | | |
| Cr | 18.27 | 18.44 | 16.35 | 16.64 | 22.09 | 22.98 | 21.40 | 21.97 | | | | <0.005 | | 0.01 |
| Cu | | 0.19 | 0.27 | 0.29 | 1.79 | 1.80 | | | 99.95 MIN. | | 90.61 | | 69.20 | BAL. |
| Fe | BAL. | BAL. | BAL. | BAL. | 30.41 | 28.09 | 3.80 | 4.42 | | | 2.44 | 2.56 | 0.54 | 0.55 |
| Mg | | | | | | | | | | | | | | |
| Mn | 1.46 | 1.38 | 1.58 | 1.56 | 0.35 | 0.33 | 0.12 | 0.13 | | | <0.01 | 0.02 | 0.57 | 0.62 |
| Mo | | 0.15 | 2.07 | 2.16 | 3.21 | 3.56 | 13.60 | 14.25 | | | | | | |
| N | 0.07 | 0.07 | 0.06 | 0.06 | | | | | | | | | | |
| Ni | 9.14 | 9.00 | 10.04 | 10.43 | 41.06 | 41.76 | BAL. | BAL. | | | 0.03 | 0.02 | 29.57 | 29.85 |
| P | 0.026 | 0.024 | 0.026 | .025 | | 0.008 | 0.008 | 0.005 | ~~0.001~~ | 0.001 | 0.006 | 0.001 | 0.004 | 0.001 |
| Pb | | | | | | | | | | | <0.01 | 0.008 | 0.010 | 0.004 |
| S | 0.005 | 0.005 | 0.018 | 0.014 | <0.001 | 0.003 | <0.002 | 0.002 | ~~0.002~~ | 0.002 | | 0.001 | 0.006 | 0.011 |
| Sb | | | | | | | | | | | | <0.01 | | <0.01 |
| Si | 0.47 | 0.44 | 0.49 | .5 | 0.19 | 0.13 | 0.03 | 0.06 | | | <0.01 | 0.02 | | 0.01 |
| Sn | | | | | | | | | | | 0.27 | 0.29 | | |
| Ta | | | | | | | | | | | | | | |
| Ti | | 0.01 | | | 0.82 | 0.93 | | <0.01 | | | | | | |
| V | | 0.05 | | | | 0.04 | 0.15 | 0.16 | | | | | | |
| W | | | | | | | 3.00 | 2.98 | | | | | | |
| O | | | | | | | | | ~~0.0041~~ | 0.0041 | | | | |

N. Sridhar
4/27/94



4/27/94 Chemical Analysis of 0.125" Thick sheet of Type 316L SS and Alloy 825

chem analysis of 0.125 " sheets

| Alloy | Heat No. | Analysis Source | C | Cr | Cu | Fe | Mn | Mo | Ni | P | S | Si | N | Ti |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Type 316L | 853252 | Vendor | 0.019 | 16.3 | | Bal. | 1.9 | 2.13 | 10.18 | 0.03 | 0.001 | 0.5 | 0.03 | |
| Type 316L | 853252 | Kawin | 0.02 | 16.36 | 0.4 | 67.5 | | 2.23 | 10.77 | | | 0.51 | | |
| Alloy 825 | HH7004FK | Vendor | 0.01 | 22.07 | 1.72 | 29.69 | 0.4 | 3.49 | 41.22 | | 0.001 | 0.29 | | 1.02 |
| Alloy 825 | HH7004FK | Kawin | 0.006 | 22.3 | 1.78 | 28.5 | | 3.68 | 41.96 | | | 0.28 | | 0.96 |

N. Sridhar
4/27/94

4/27/94 Chemical Analysis of Alloy 825 weld wire for electrochemical connections.

This is a 0.045" dia. x 36" long wire of Incoloy alloy Filler Metal 65.

Heat No. HH5230F.

| Element | Composition, wt. %: INCO | Composition, wt. %: Kawin |
|---|---|---|
| C | 0.01 | 0.022 |
| Cr | 22.16 | 21.99 |
| Cu | 1.72 | – |
| Fe | 31.31 | 30.70 |
| Ni | 40.85 | 41.40 |
| Mn | 0.32 | – |
| Mo | 2.70 | 2.70 |
| S | 0.001 | – |
| Ti | 0.79 | 0.75 |
| P | 0.018 | – |
| Si | 0.09 | 0.12 |

N. Sridhar
4/27/94



6/10/94 Polished Samples

| Sample Id | | Sample |
|---|---|---|
| 825-SA-SEN 100-750 | - | SA 1200°C for 10min Sensitized 750°C - 100hr |
| 825 MA 100-750 | - | millannealed 750°C for 100hr HH4371FC |
| 825 → 20Cr | - | 825 HH4371FG -20Cr |

- made stock solution by adding ~~[illegible]~~ gm 6/10/94
  day prepared 6/10/94
  950 ml ~~37%~~ gm 6/10/94 37% HCl Lot No. 943809
  50 g Oxalic acid Lot No. 926502

electrochemical etch done with 125 ml stock solution and ~~[illegible]~~5 ml water · voltage and current reduced
Sample area 5cm², 250 mA, 1 Volt, ~~Fe~~ gm 6/10/94
Voltage + Current set with Fluke 80508 Id# 5005110
+ SA 1200-10 ptxl 25 sec - 2 volts 500 mA 5cm² area
~~-80-5~~ gm 6/10/94

Procedure - Run current at approximately 100 mA/cm²
- voltage should be set at 2 volts
- Voltage can be set at 1 3/4 volts for SEN samples

Sensitized sample was not etched with stock solution
Methanol $H_2SO_4$ solution was used from P.29 with 4 1/2 V and lamp.

Jerry Mellen 6/13/94

Jerry Mellen 6/13/94

-6/13/94 Polished Samples

attempted Electropolishing samples using methods described on page 59.

Samples 1, 2, 3 were polished mechanically using 1000, 2000 grit sand paper then 3µ diamond paste which was followed by pollishing with colloidal silica solution. This ~~exp~~ j.m 9/13/94 ~~did~~ jm 9/13/94 These method did not yeild good results, scratches remained in the sample through the electropolishing step. These steps differed from the process described on pages 27-30.

| Sample # | Sample description |
|---|---|
| 1 | 825 46cr HH 4371F6-46cr |
| 2 | 825 36cr HH 4371F6-36cr |
| 3 | 825 61cr HH 4371F6-61cr |

The samples were etched for various times; the best results can from ~~4~~ jm 9/13/94 sample 3 which was etched for 40 seconds at 2 volts and 120 ~~mA~~ jm 9/13/94 mA. Sample 1 and 2 were etched under the same conditions for 1:30 and 1:400 respectivily.

Jerry Mehlen 6/13/94
jm 6/13/94

Jerry Mehlen 6/13/94
jm 6/13/94



~~9/14/94~~ jm 6/14/94

~~9~~ 6/14/94 Polished samples

| Sample # | Sample description |
|---|---|
| 4 | HH4371F6 Hot rolled to 0.285 in., mill Annealed |
| 5 | HH4371F6 Mill anealed, Hot rolled to 0.285 in, Polish surface perpendicular to roll direction, |
| ~~6~~ jm 6/14/94 6 | HH4371F6 mill anealed, Hot rolled to 0.285 in. Thick polished surface perellel to roll direction, |

Polished use instructions on P. 27-30 until 600 grit, after 600 grit 1000 and 2000 grit sand paper were used the pressure per sample was 3 lb for the 1000 grit and 2 Lb for the 2000 grit paper, extra care was taken in keeping the samples clean to prevent cross-contamination.

Jerry Mehlen 6/14/94

6/15/94 Polished Sample

- Obtained suitable results using 1000 and 2000 grit paper
- 1000 grit 3Lb per sample 4 min
- 2000 grit 2Lb per sample 4 min
- 3µ diamond paste 12 min 4 ~~4~~ jm 6/17/94 Lbs per sample first 9 min 3Lbs per sample last 3 min
- Colloidal silica 6 min ~~xx~~ jm 6/15/94 4lbs per sample

Jerry Mehlen 6/15/94

6/16/94 Electrochemically polished samples

Procedure

Made polishing solution by adding
170 ml methol alcohol (methanol) Fisher chemical Lot # 933242
30 ml $H_2SO_4$ sulfuric acid Fisher chemical Lot # 940182
-Sample were emersed in solution and ~~etche~~ polished for 4 min
Voltage - 3V Current - 1A OD 9/15/2000
- microstruct was exposed.

Jerry Mehlen 6/16/94

Jerry Mehlen 6/17/94

6/17/94 . polished Samples

| Sample # | | Sample Identification |
|---|---|---|
| 7 | 825 | Hot rolled polish parallel HH4371FG |
| 8 | 825 | Hot rolled polished perpendicular HH4371FG |

Procedures

- Followed Procedures on p.27-30
- electropolish using Solution p61 for 4min

| Sample # | | Sample Identification |
|---|---|---|
| 9 | 825 | 20% Cold rolled HH4371FG Perp. Polish |
| 10 | 825 | 20% Cold rolled HH4371FG Parallel Polish |
| 11 | 825 | 20% cold rolled 100 hours - 750°C HH4371FG |

Procedures

- Followed Procedures p.27-30
- electropolish using solution p.61 for 4min sample 9,10,11
- etched Sample using Stock (JM 6/20/94) Solution from page 59
- Sample 9 was etched for 5 seconds at 120mA and 2volts. Sample 10 was etched for 5 seconds at 2volts 205mA. Sample 11 was not etched.

results

etching produced poor results, Sample were polished again mechanical and then electropolished for 5min.
- Bonding exposed as when as grain boundries
- Bonding made grain boundries difficult to expose.

Jerry Imehlen 6/20/94



6/27/94

Jerry Imehlen 6/27/94

- Metallographic technique on page 27 was follow with two exceptions ~~exceptions~~ to obtain photographs (JM 12/1/94)
1. 1000 and 2000 grit Sand paper were used instead of 3μ diamond Lapping Film
2) Only electropolishing was (JM 6/27/94) used to expose microstructure.

Jerry Imehlen 6/27/94

6/27/94

no negative

Neg. #3001 0.5" plate 2150°F Hot roll +MA +61% CR 100x

Sample #3 HH4371F6.

no negative

Sample #3 N. #3002 Same as 3001 at 200x Jerry Mehalin 6/27/94



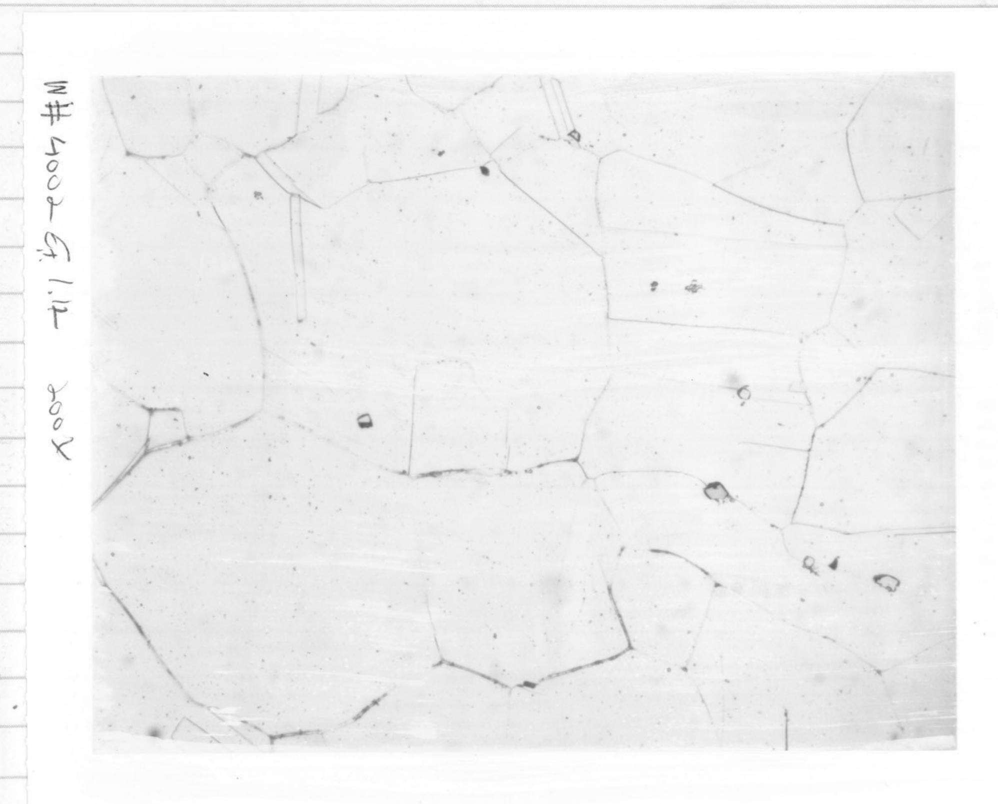
6/27/94

no negative

Neg # 4001 0.5" Plate +2150°F, Hot roll (0.3") +MA 100x

Sample # 4 HH 4371F6

no negative



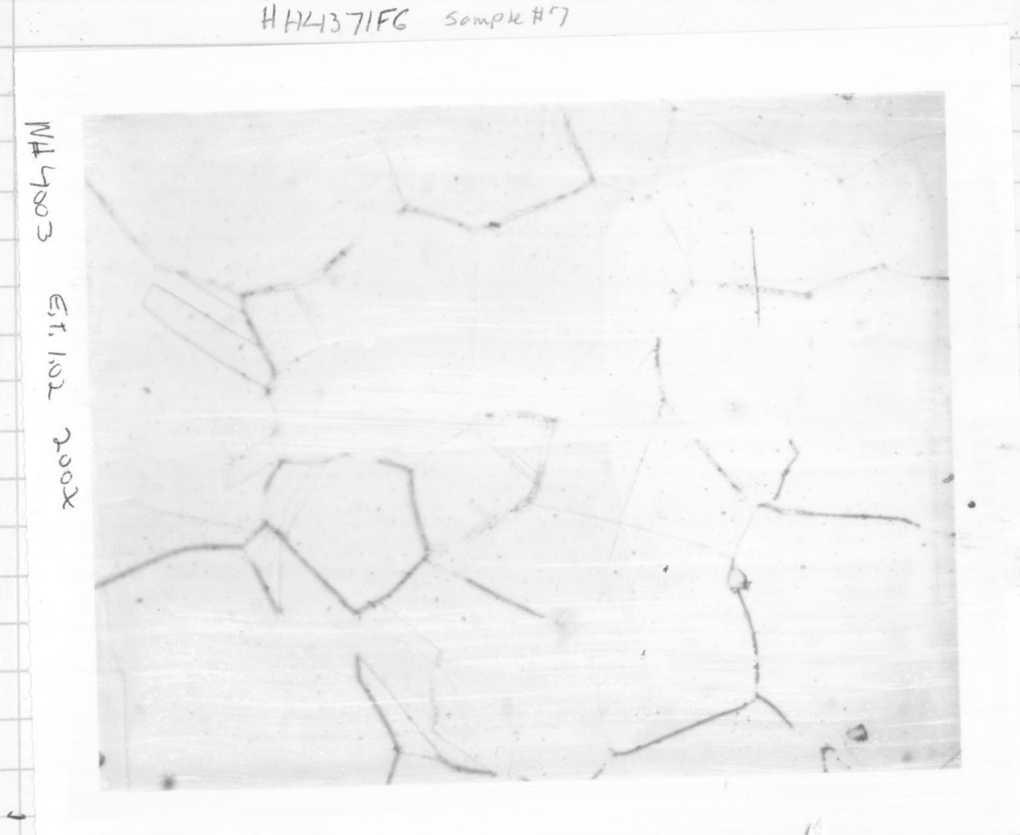
Neg #4002 0.5" plate +2150°F, Hot roll (0.3") +MA 200x

Sample #4 H4371F6

Jerry Mehalin 6/27/94


no negative

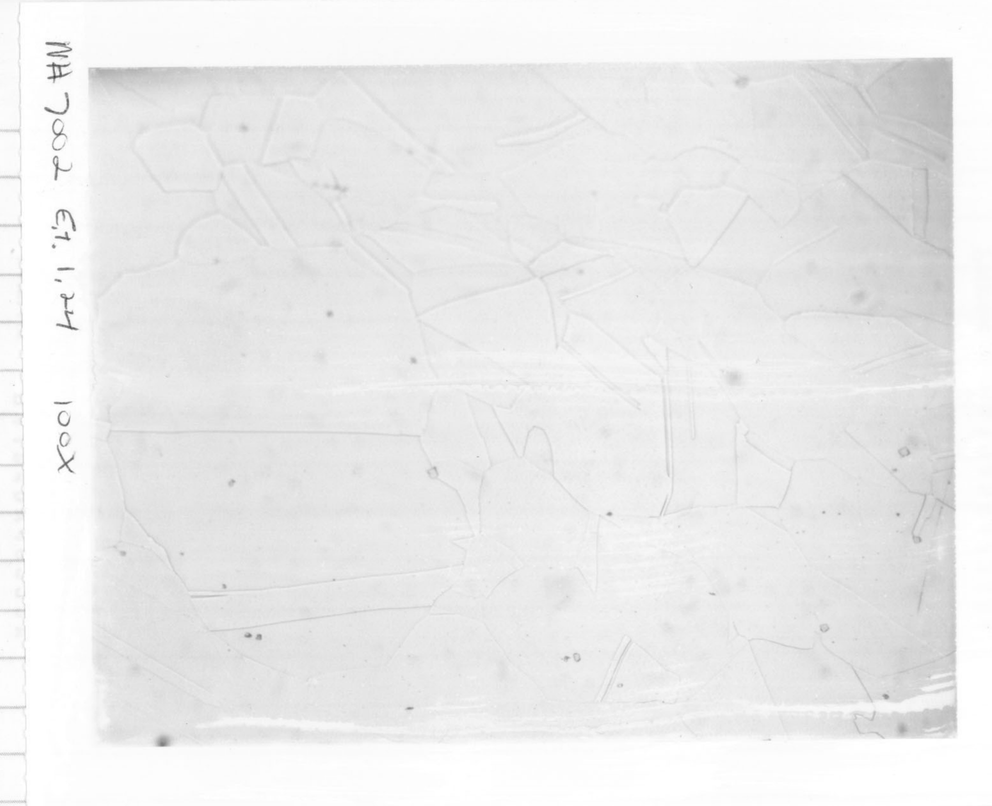
#7001 0.5" plate +2150°F Hot roll (0.3") + ~~MA~~ As Hot rolled 100X
G.m 6/27/94
HH4371FG sample #7




no negative

#4003 0.5" +2150°F Hot roll (0.3") + Mill anneal 200X
No aging HH4371FG sample #4 Jerry [signature] 6/27/94






no negative

#7002 0.5") plate +2150°F Hot roll (0.3") as Hot rolled 100X
HH4371FG sample #7

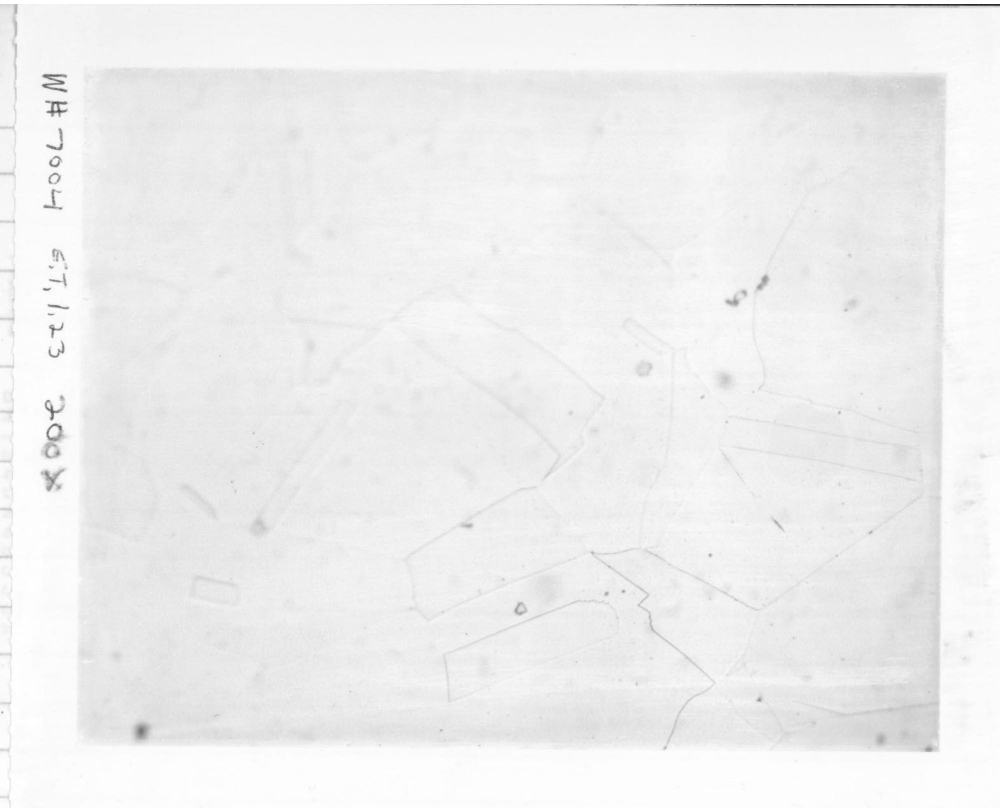

no negative

#7003 0.5" plate +2150°F Hot roll (0.3") as Hot rolled
HH4371FG sample #7

Jerry [signature] 6/27/94

6/27/94

no negative



#7004 (0.5") plate +2150°F + Hot rolled (0.3") 48 Hr.
HH4371F6 Sample #7 100X

no negative

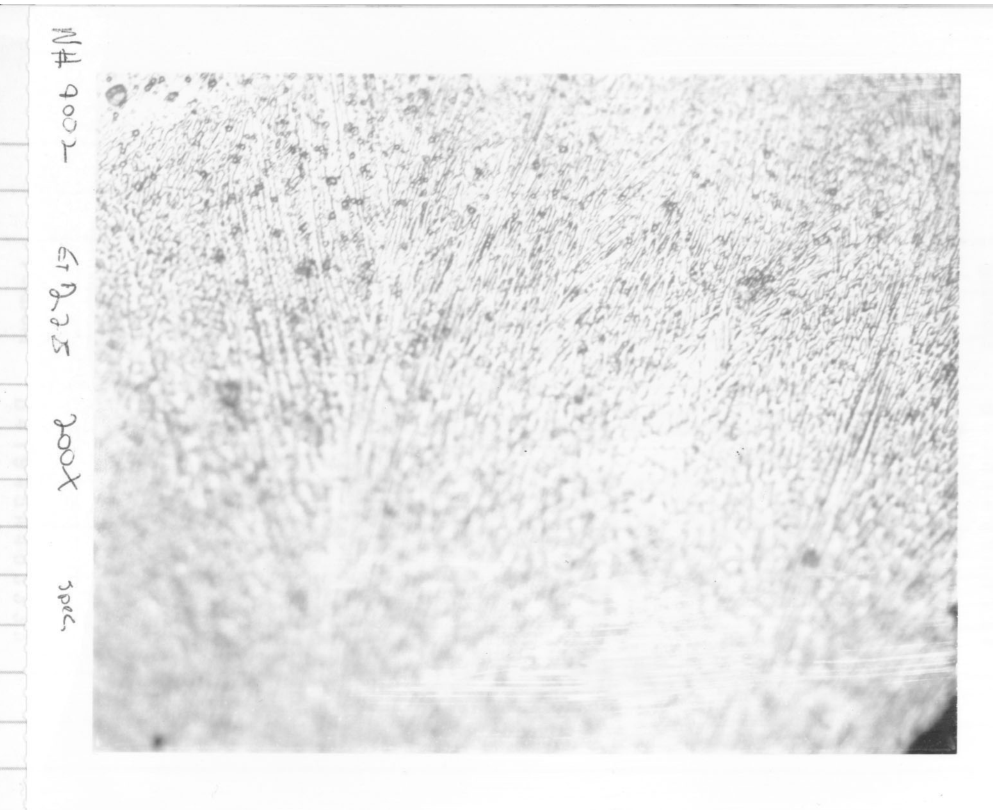
9001 MA (0.5") + cold roll at Haynes 20% cr no aging 100X
[* note! photo of Atypical microstructure in corner of Sample] HH4371F6
Sample #4

Jerry Mellen 6/27/94



6/27/94

no negative



#9002 MA (0.5") + 20% cold roll at Haynes no aging 200X
[* Note: Photo of Atypical microstructure in corner of Sample] HH4371F6
Sample #9

no negative

#9003 MA (0.5") plate 20% cold roll at Haynes no aging 100X
HH4371F6 Sample #9

Jerry Mellen 6/27/94