Development of MA957 at PNNL for Fast Reactor Application: Prior Work (to 2000)

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"Fe- Based ODS Alloys: Role and Future Applications"

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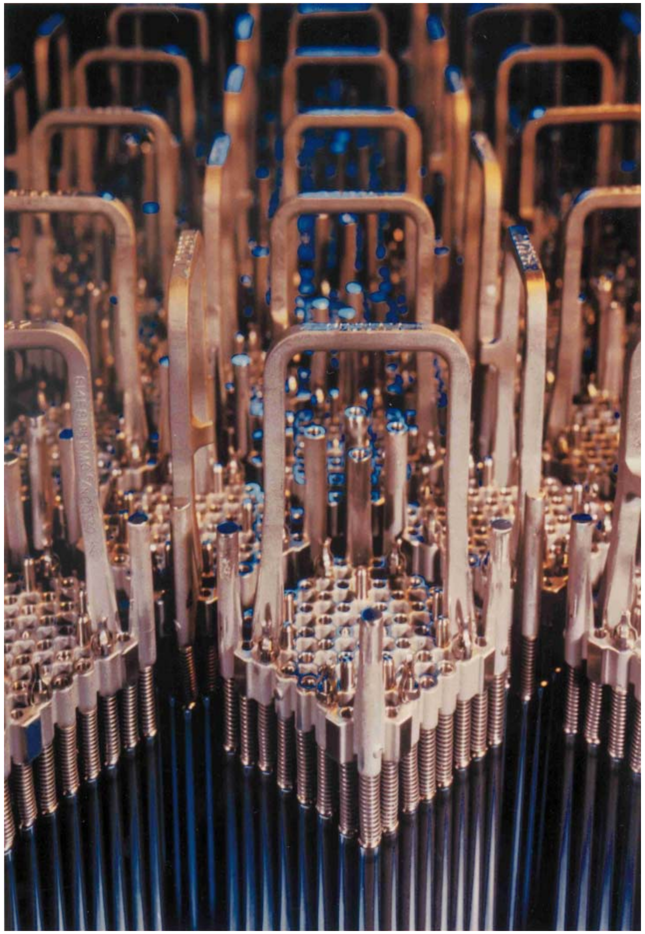
- Discussion of work performed prior 2000
  - ODS MA957 for Liquid Metal Reactor Cladding tubes
    - Supplier development - to develop multiple sources for tubing
      - Security of supply for reactor operations
    - Characterization of ODS MA 957
    - Production validation
      - NDE – not discussed
      - End closure joining – EM pulse joining was qualified – not discussed
- Where ODS may be headed
  - What may be done to speed qualification

- **Motivation:**
  - **US DOE sponsored work to develop improved fuel cladding materials for liquid fast metal breeder reactors since the early 1970s**
    - **Solution annealed 316 SS – irradiation-induced swelling limits lifetime to ½ - 1 year of use**
    - **316 SS in cold worked condition – extended lifetime to 1.5 yrs**
    - **Ferrittic/martensitic HT9 – lifetime out to 3yrs**
    - **ODS ferritic alloys – lifetimes of 5+yrs**
  - **Elevated temperature strength**
    - **Reactor temperatures less than 800°C**
    - **Non-recrystallized ODS**



DOE Project

- Objective (series of studies):
  - Develop processing schedules for clad tube production
  - Determine the properties of MA957 in the unirradiated and irradiated conditions
  - Understand the behavior of MA957 with respect to its application as a cladding material for a fast breeder reactor
- Multi-year/Multi-investigator project
  - PNNL
  - Westinghouse Hanford Co.
    - Vendors: Inco, Cartech, STC, PNC
    - New names: Special Metals, Veridiam
  - General Electric Co.
  - University of Missouri-Rolla

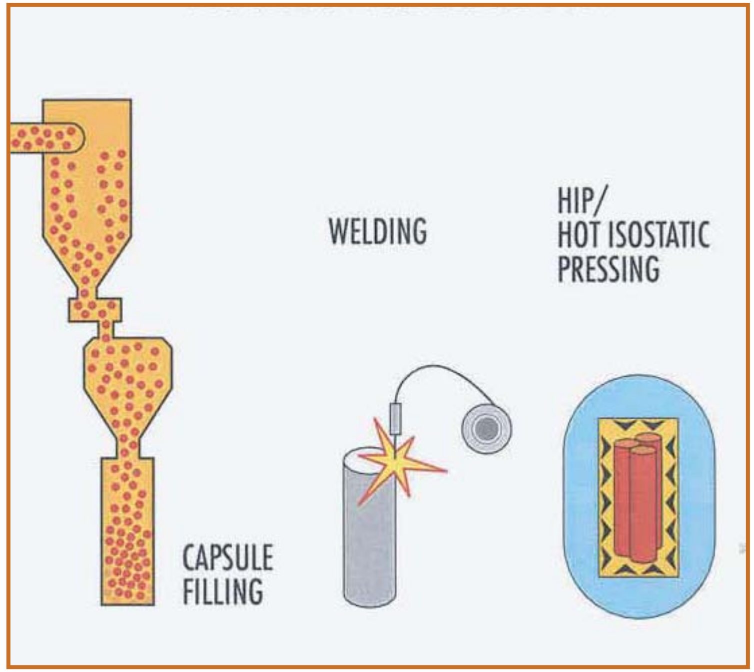
- **MA 957: ferritic alloy strengthened by a dispersion of Y_2O_3**
  - **Base composition: Fe-14%Cr-1%Ti-0.3%Mo-0.25% Y_2O_3 (weight percent basis)**

ELEMENT	WEIGHT PERCENTAGE		
	AVERAGE	RANGE	NOMINAL
Cr	13.87	13.49-14.19	14
Ti	1.05	0.95-1.38	0.9
Mo	0.30	0.28-0.32	0.3
$Y(Y_2O_3)$	0.22	0.19-0.28	0.25
0	(a)	0.006-0.240	-
C	0.014	0.012-0.017	-
Mn	(b)	0.05-0.12	-
Si	0.04	0.02-0.07	-
P	(c)	0.004-0.030	-
Ni	0.13	0.10-0.15	-
Al	0.10	0.055-0.17	-
S	0.006	0.004-0.006	-
Fe	Bal.	Bal.	Bal.

- Al, O, C picked up from starting powders and milling
- Ti was added for improved oxidation resistance and to minimize Cr volatilization
- Mo was added to improve room temperature ductility and strength
- Variability was observed
  - To be expected in short run P/M

Materials Studied: General Fabrication

HIGH ENERGY MILLING



Basic technologies used for this project:

- HIP/Extrusion by INCO to produce bars that were drilled to produce hollows(Wiggin Alloys Ltd.)
- Drawing over mandrel(plug and traveling rod)
- Hot drawing operation

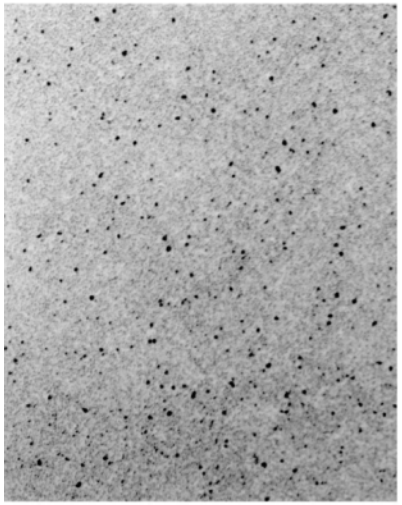
Other techniques looked at later but not tested

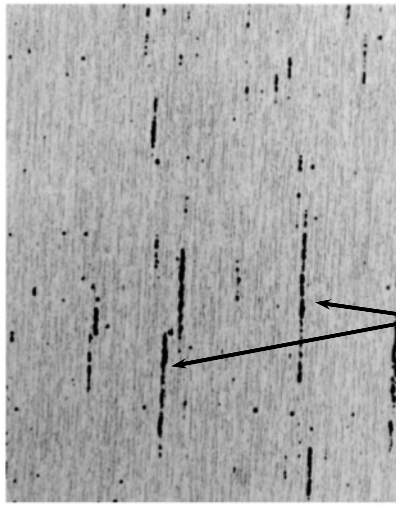
- Pilger
- Hydrostatic extrusion

Materials Studied: Extruded and Swaged Bar Stock

Optical Micrographs:

TRANSVERSE | **LONGITUDINAL**



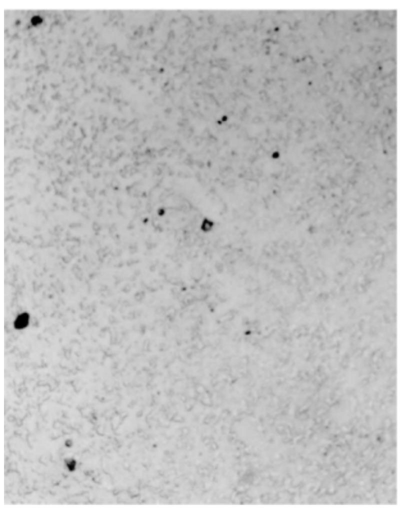


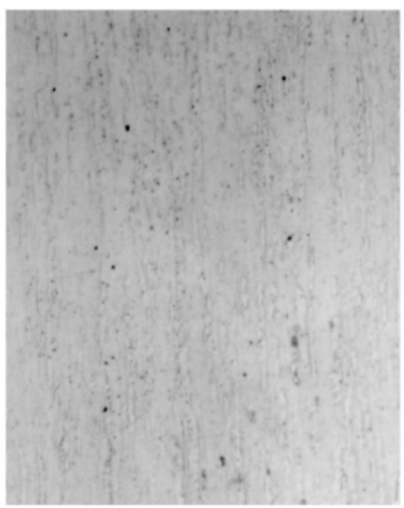
100x

Al_2O_3 stringers

Grains are equiaxed in this direction

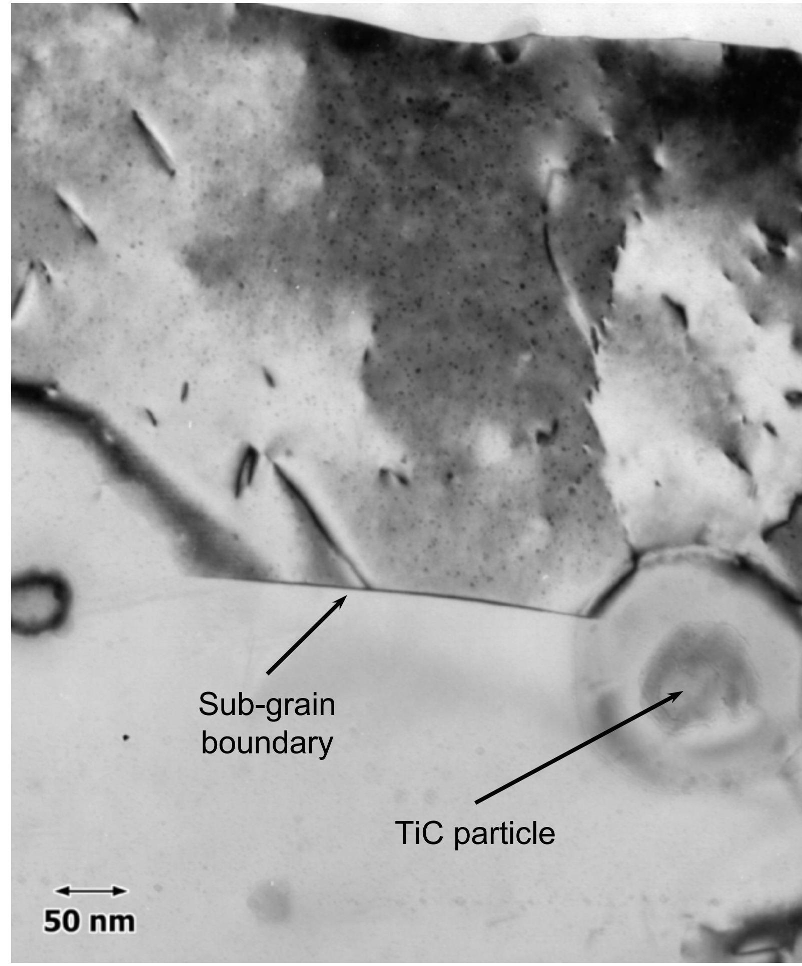
Grains are elogated in this direction





1000x

TEM Micrograph:



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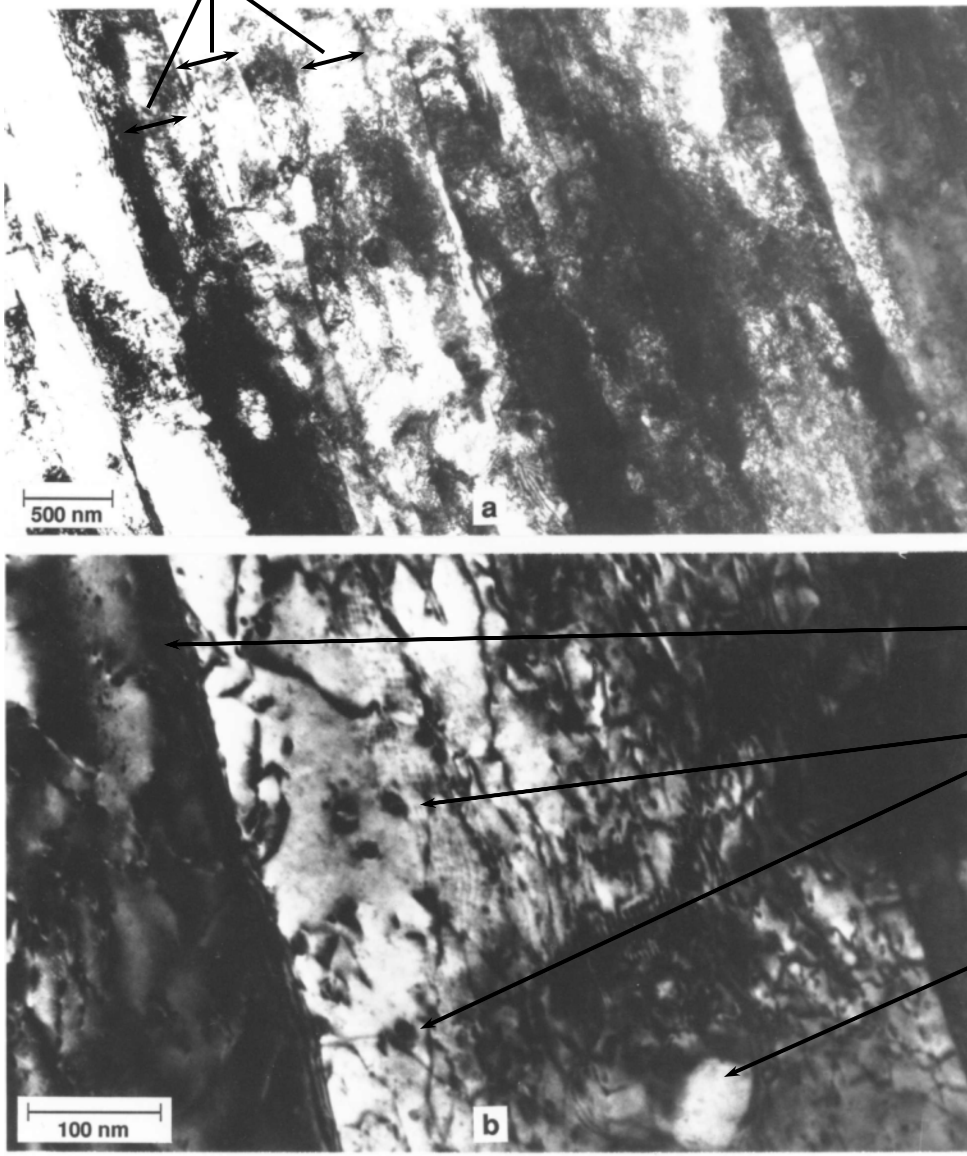
Materials Studied: Extruded/Drawn Tubing



Carpenter Technology Corporation (Cartech) – MA 957 tubing

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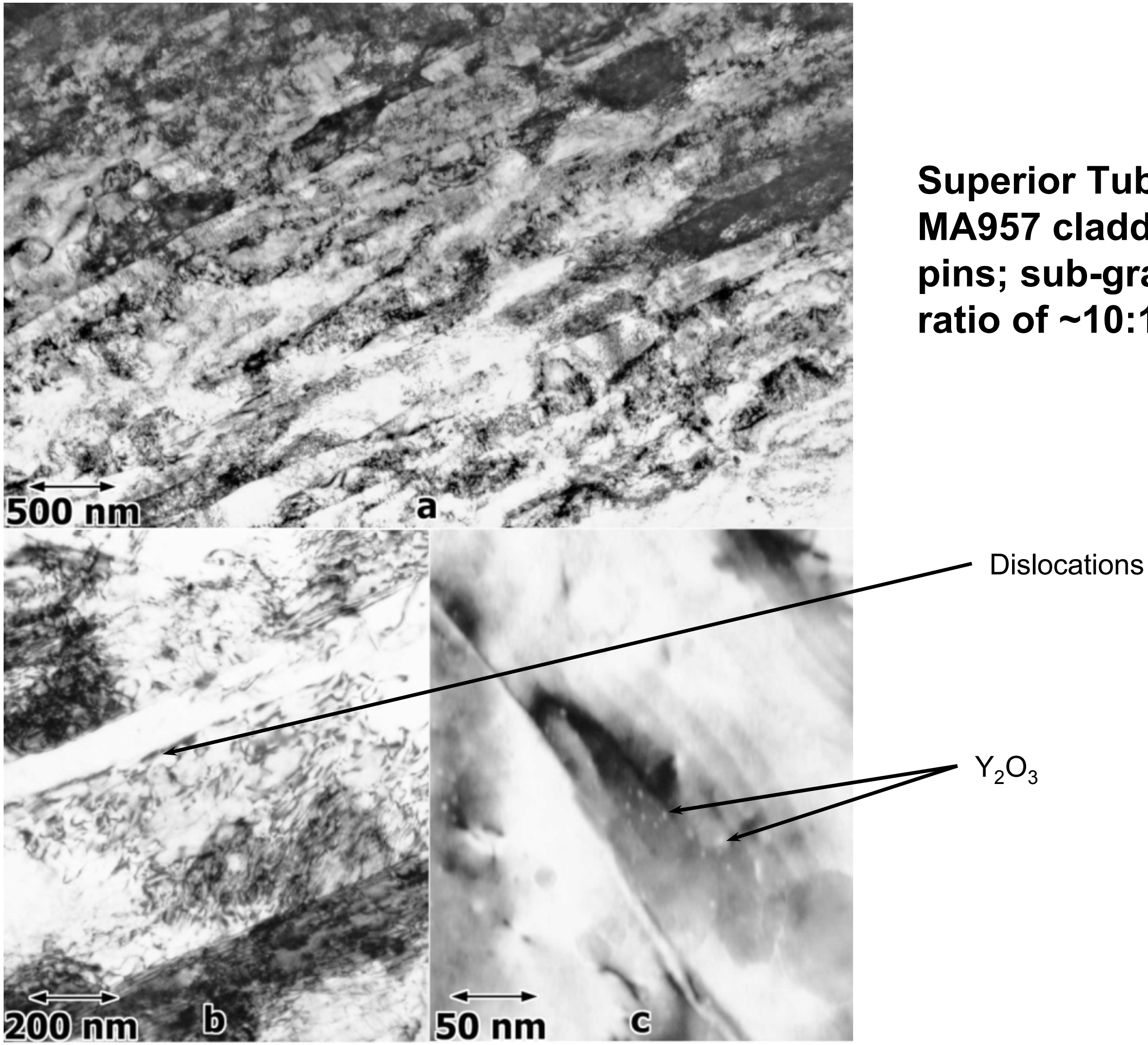
Materials Studied: Extruded/Drawn Tubing



Superior Tube Company (STC) – MA957 cladding for EBR-II fuel pins; sub-grains have an aspect ratio of ~10:1.

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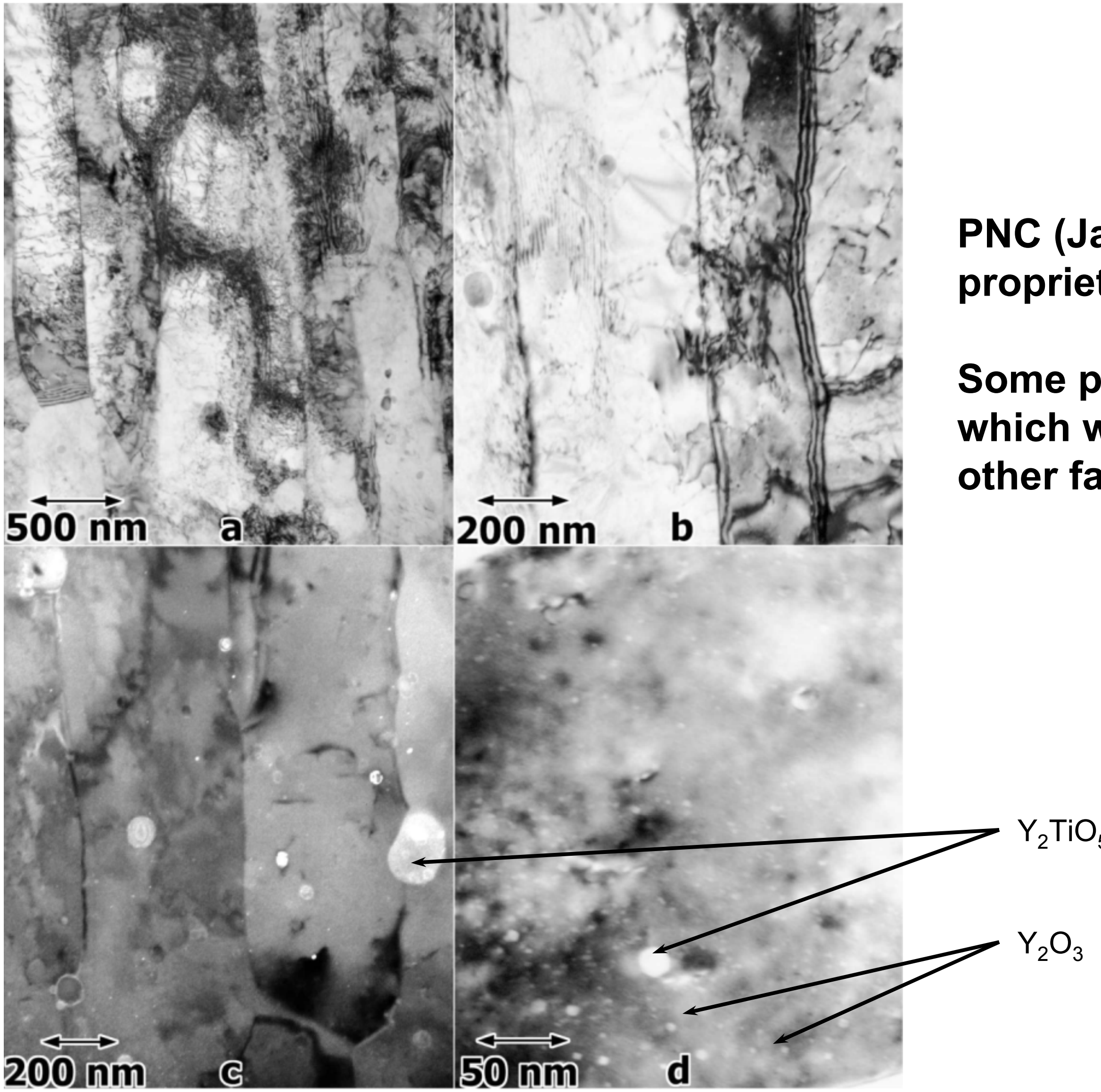
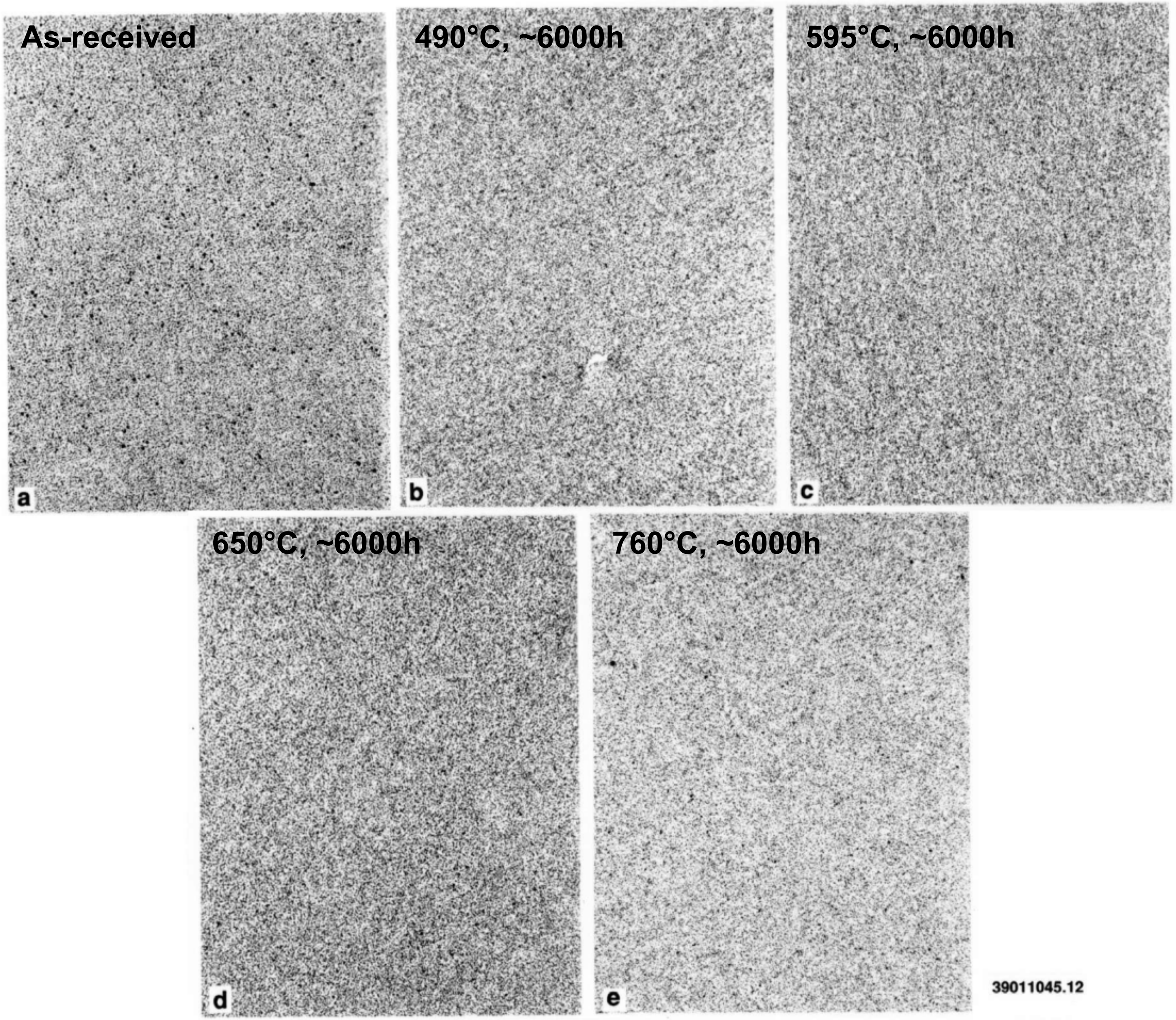


PNC (Japan) – tubing formed by a proprietary hot drawing process.

Some precipitates where coherent which was not observed with other fabrication methods

Aging/Recrystallization Behavior



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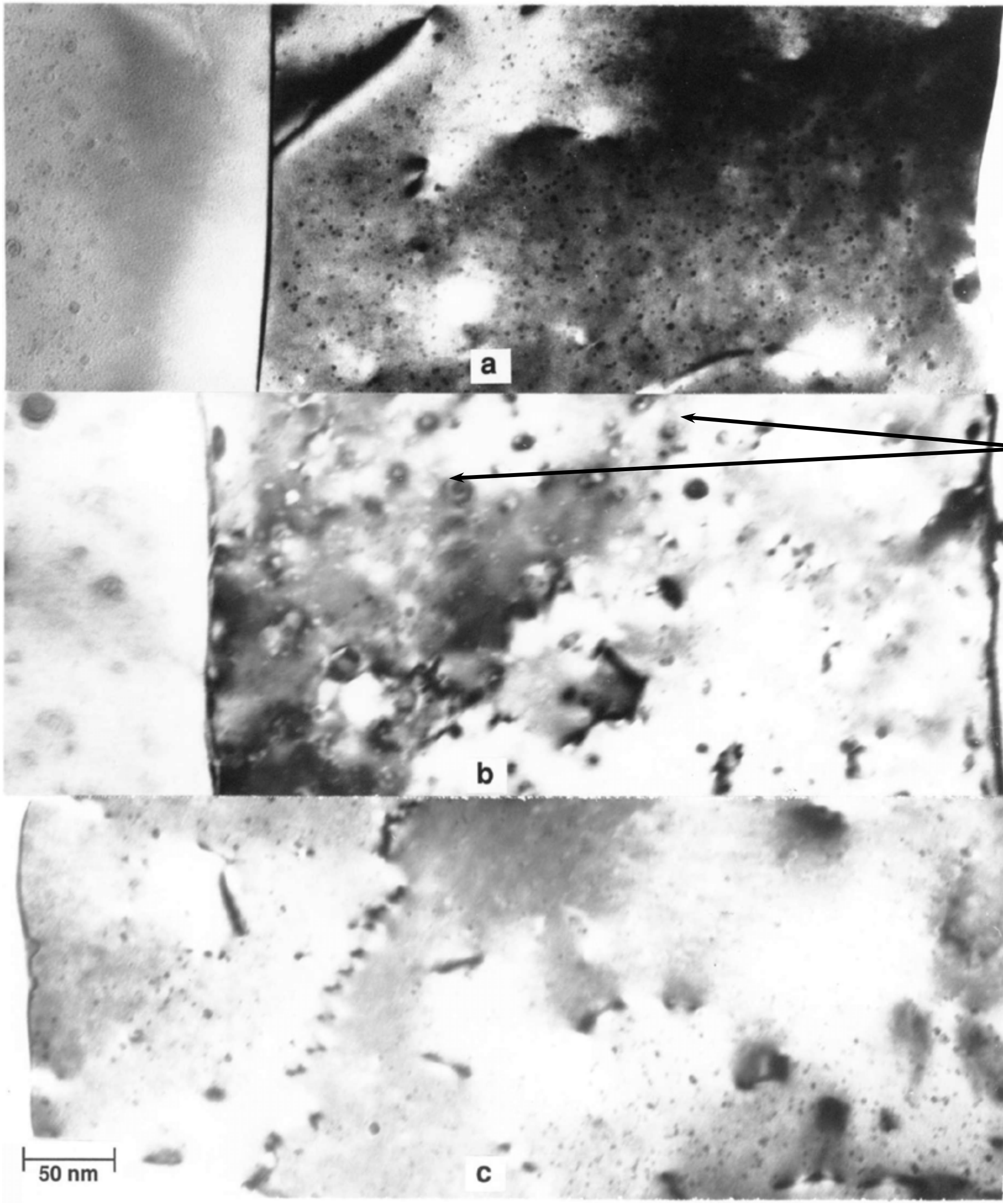
Aging/Recrystallization Behavior



As-received

a' phase (Cr-rich)

490°C, ~6000h

760°C, ~6000h

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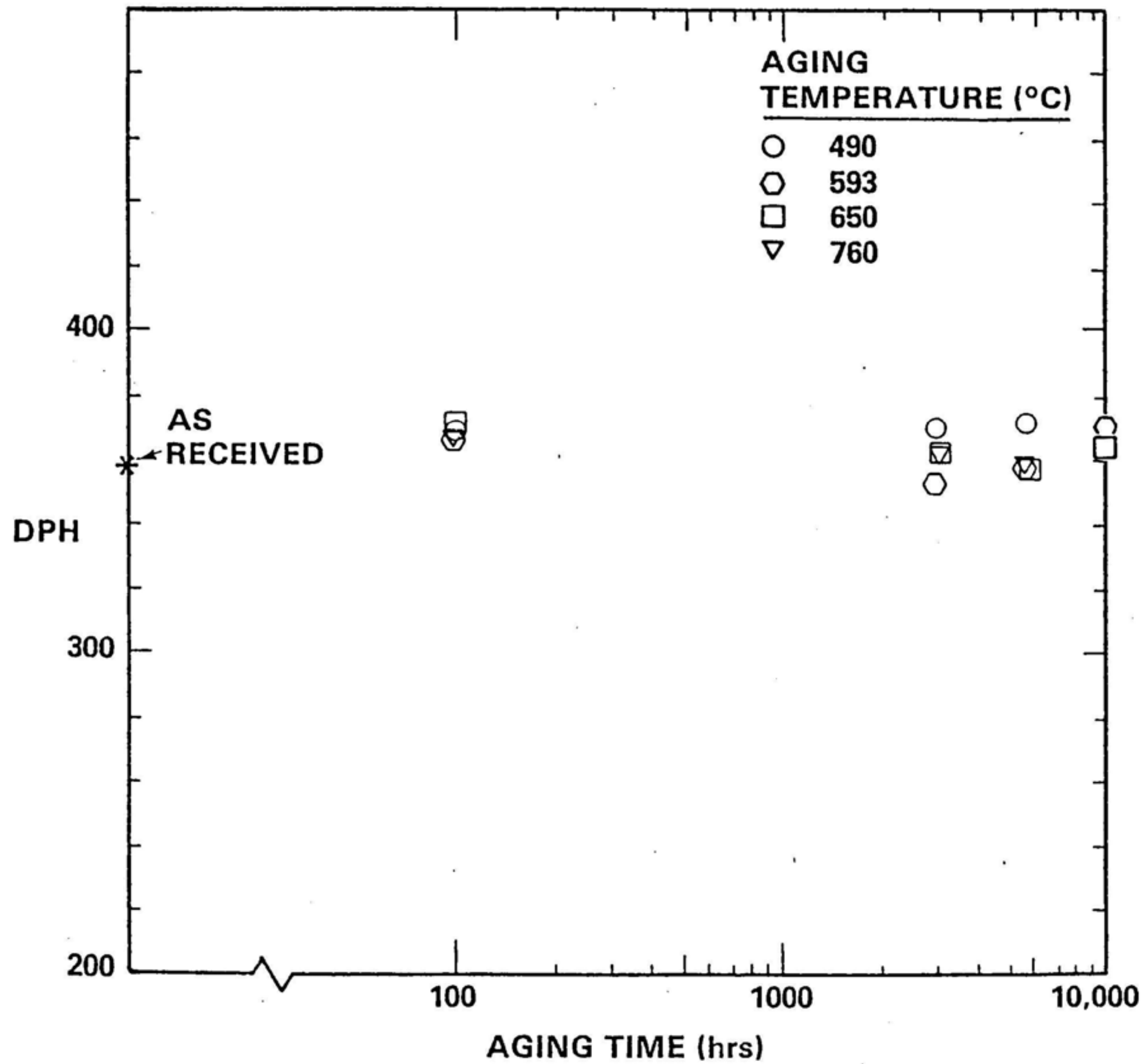
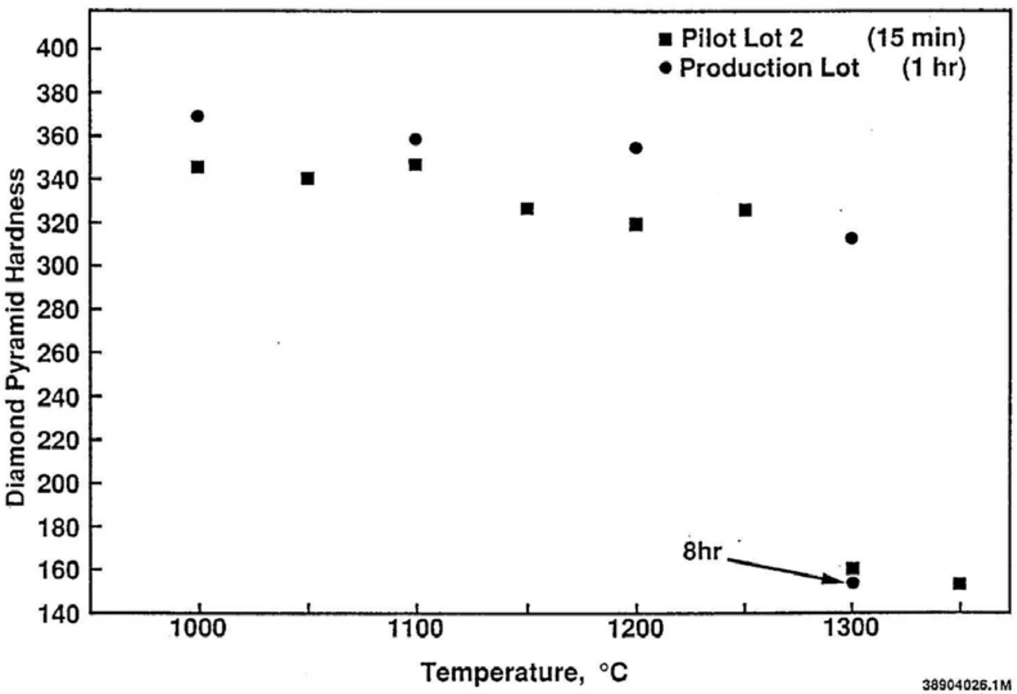
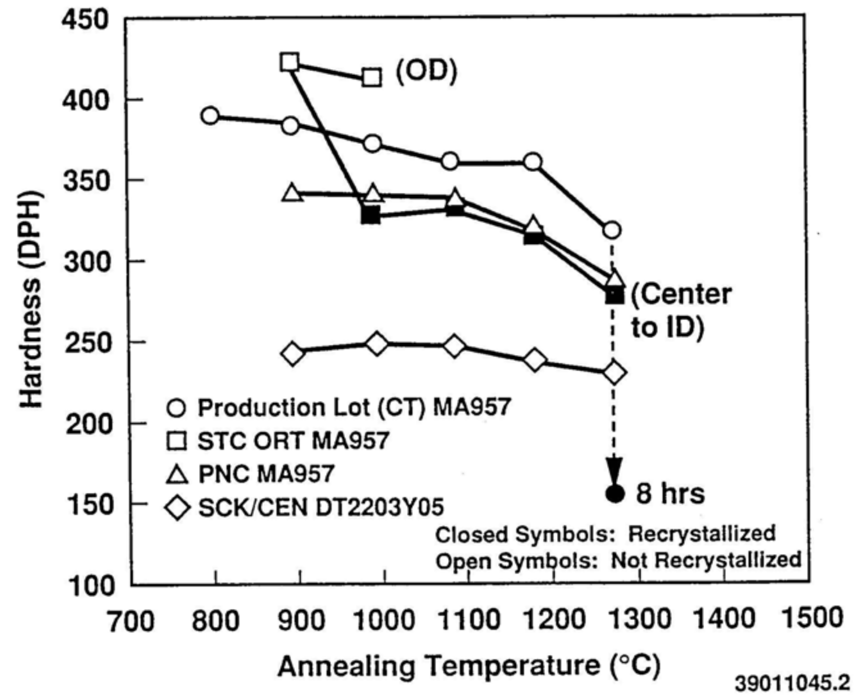
Aging/Recrystallization Behavior



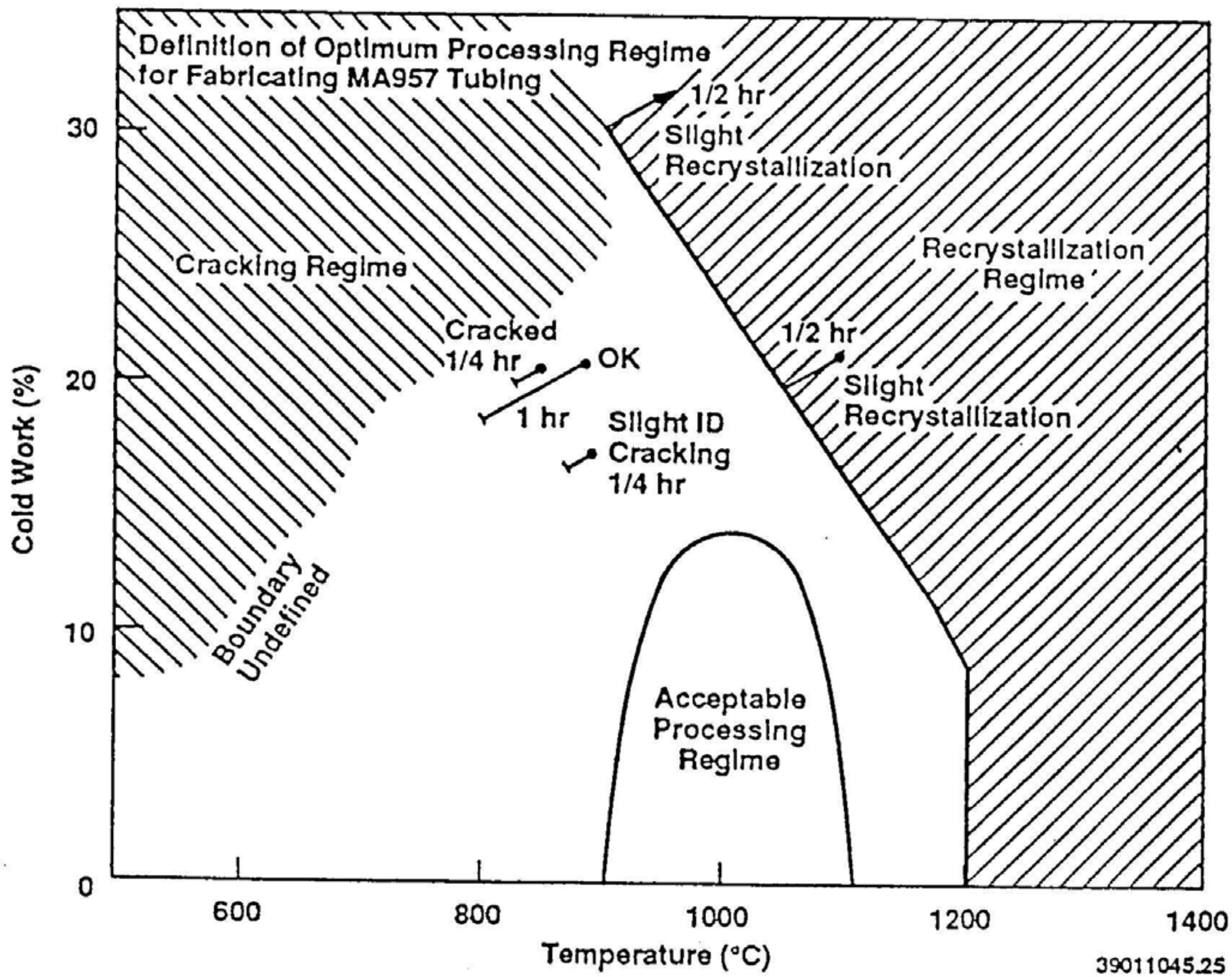
No change in hardness out to 10,000h at each temperature





Recrystallization at ~1300°C for rod stock – measurable drop in hardness

Workable processing regime for fabrication of MA957 tubing found in this study:



Cracking/recrystallization is avoided if cold work is held to ~15% (~1000°C annealing temperature)

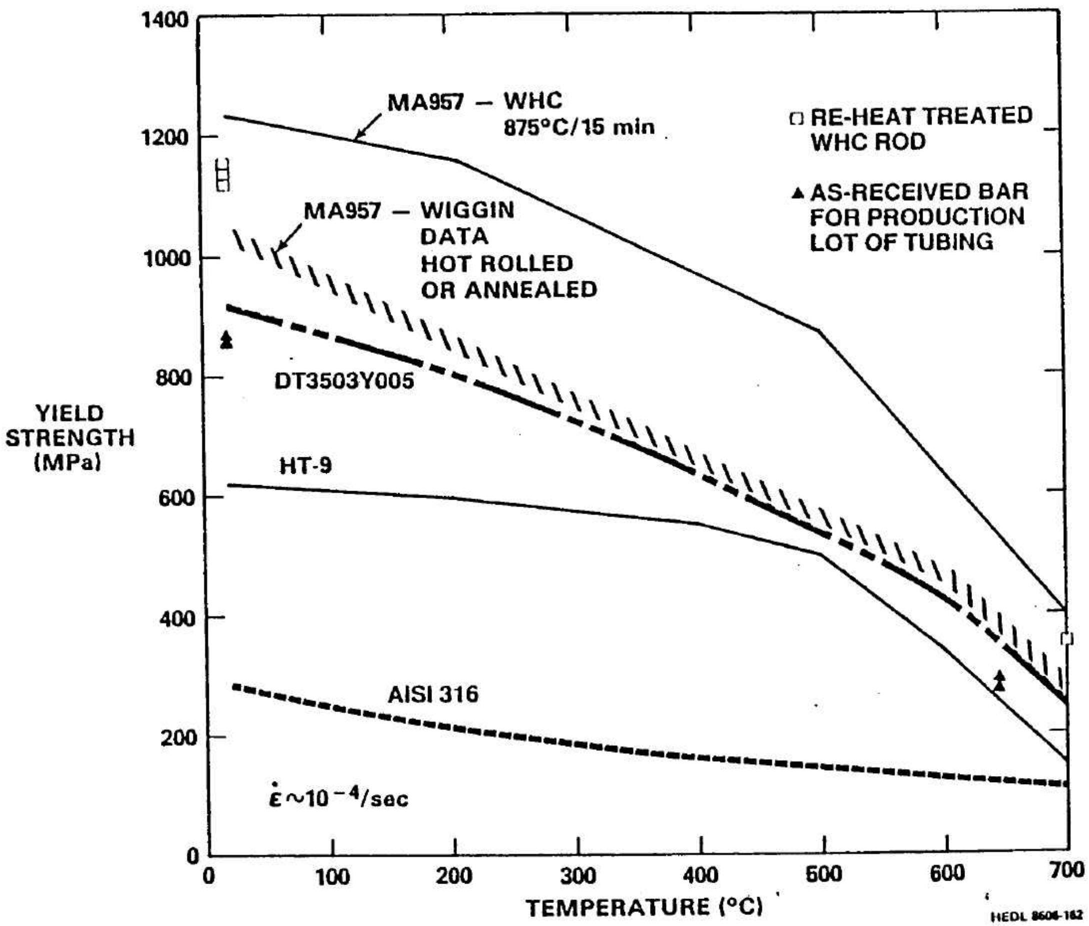
- Two factors controlled the selection of the final fabrication path:
  - Need to minimize the cost of tubing fabrication (by minimizing the number of reductions by using the largest possible reduction/pass)
  - Need to maintain the as-received microstructure (which limits secondary work temperatures)
- WHC tubing fabrication results:
  - Successful swaging at 1050°C, but recrystallization occurred
  - Reduction of a tube hollow with 800°C intermediate anneals resulted in cracking (thus, the optimum stress relief temperature was determined to be between 825 and 1050°C)
  - On the above basis, employed 15% reductions and 825°C anneals – employed multiple passes (25) on pre-drilled rod to reduce from 0.875" (OD) x 0.476" (ID) to 0.230" (OD) x 0.200" (ID) without recrystallization.
- STC tubing fabrication results (by conventional practice):
  - Employed rod drawing and two-roll de-rodding
  - Experienced some cracking until the nose and point was heated to red heat
  - Used 17 rod draws to fabricate 8' of 0.270" (OD) x 0.226" (ID) tubing

Fabrication: Additional Findings

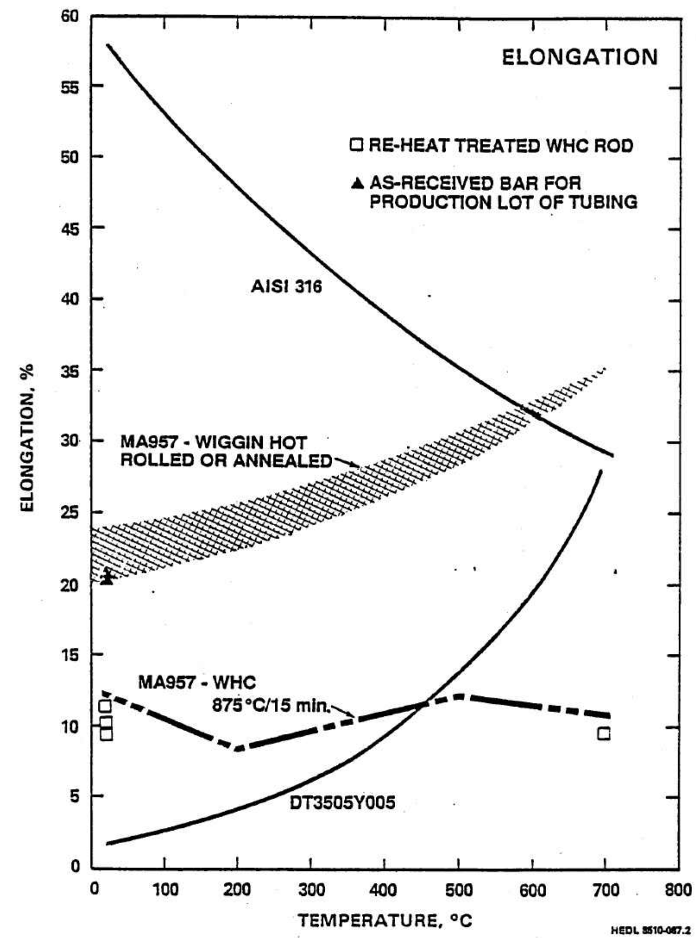
- **Cartech tubing fabrication results:**
  - **Employed plug drawing with 875°C intermediate anneals**
  - **Had significant processing difficulties (including transverse cracking) – heating to 925°C did not produce the desired softening**
  - **Attempts to machine away ID and OD cracks and continue drawing ended up with split tubes**
  - **Ultimately ended up gun drilling tubes**
  - **Also had later success with rod drawing (similar to STC)**
- **Alternative processes: obvious difficulties with conventional drawing methods are related to limited tensile ductility. Alternative processes focused on hydrostatic metal working techniques:**
  - **STC – carried out pilgering on tube hollows; achieved 19 – 39% reductions with minimal problems (8 passes to achieve desired final reduction dimensions)**
  - **UMR – employed hydrostatic extrusion; only a small amount of material made, but technique showed some promise (area reductions of 40 - ~50% achievable in a single pass)**
  - **Proprietary processes employed in Japan – little information; understood to be hot worked at 400 – 800°C**

Properties: Strength and Ductility

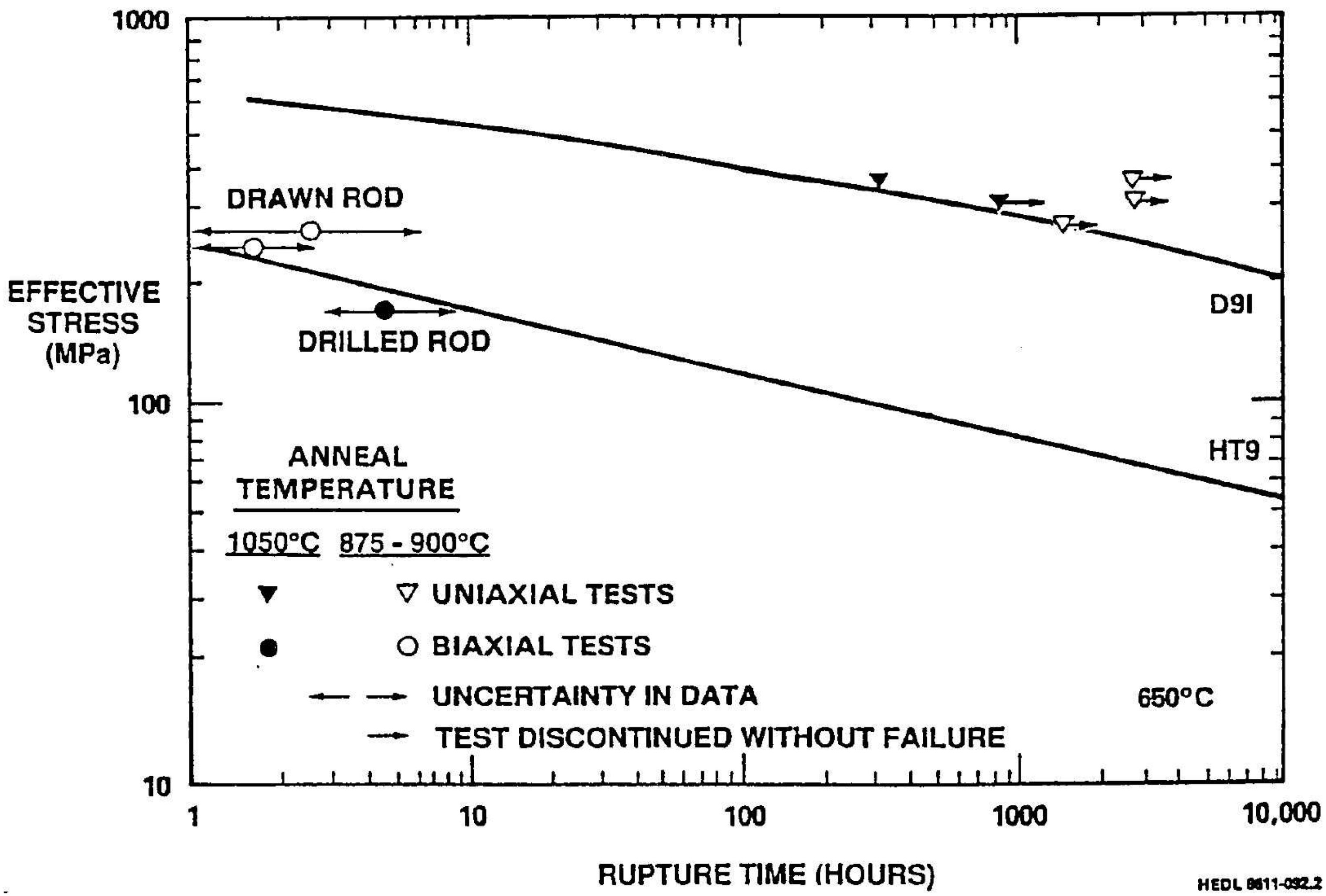
Yield Strength



Ductility



Properties: Stress Rupture Behavior



Irradiation

- A less than 20% increase in yield strength was observed
  - No change in irradiation above 500oC
  - 25% decrease in ductility
- Creep was less than HT-9
- Diametrical changes were minimal under conditions tested

Conclusions

- Primary focus of the work – developing MA957 tubing for irradiation testing/ characterization and out-of-pile characterization
  - Lead by WHC
  - Materials obtained from INCO
  - Additional tubing fabrication: WHC, STC, Cartech, UMR, PNC
- Issues in forming tubing
  - Required large number of passes to reduce to proper tubing size
  - Difficulty controlling recrystallization during hot forming/working
- Suspect much of the fabrication problems can be overcome by developing proper fabrication sequences
- Properties
  - High temperature yield strengths far exceed those of HT9
  - Rupture strength trends higher than HT9 or D91
  - Relatively low ductility (particularly at room temperature)

- ODS materials still consistently appear in the projected needs for US advanced nuclear reactor materials needs
  - Attractive combination of elevated temperature strength and irradiation behavior

- ODS materials still carry the inherent probabilistic characteristics of P/M processing and in the 1970's this was not as accepted and P/M materials were rejected for wrought materials even when lower performing
  - Probabilistic nature better understood – only in the last 5 years P/M materials are used on commercial aircraft
- Tube production methods have improved since the 1970 and 1980s
  - Newer generation machines and tool designs produce difficult to form materials at cost effective rates

Recent PNNL activities in ODS for Nuclear Applications

- **Presentations made by Glenn Grant**
- **Small internally funded activity to see if new materials characterization methods can be used to predict the probabilistic nature of ODS and promote acceptance**
  - **Based on testing methods used to qualify:**
    - **Weld processes and advanced steels for automobiles**
    - **Titanium tubing for aircraft hydraulic systems**
    - **Zirconium tubing for LWR nuclear fuel clad**

Digital Imaging of strain to predict variability

Rolling of MA956