

Creep-Testing Foils and Sheets of Alloy 625 for Microturbine Recuperators

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Background

Temperature and water vapor enhanced oxidation are the primary factors affecting degradation, with stress and creep becoming factors at higher temperatures and longer times

- **Temperature**
- **Environment** (combustion gases can lead to corrosion)
- **Mechanical Stress** (pressure differential can induce creep deformation)

To Achieve DOE Advanced Microturbines Program Goal (efficiency >40%), Microturbines will operate at higher temperatures and stresses.

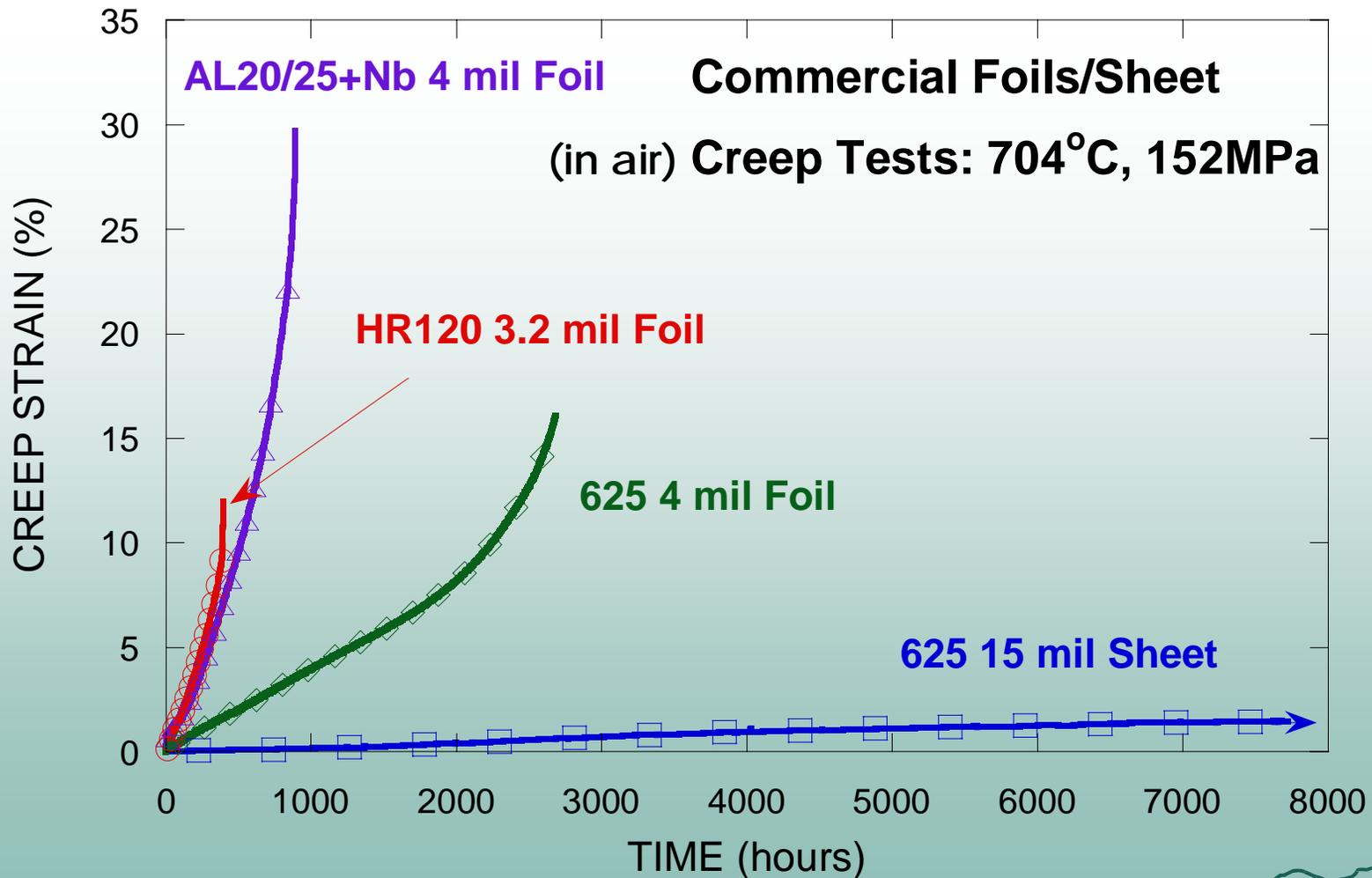
- **Resistance to creep critical for useful component life**

Advanced Austenitic Commercial Sheet and Foils Have Been Evaluated For Use In Recuperators (Alloy Compositions (wt.%))

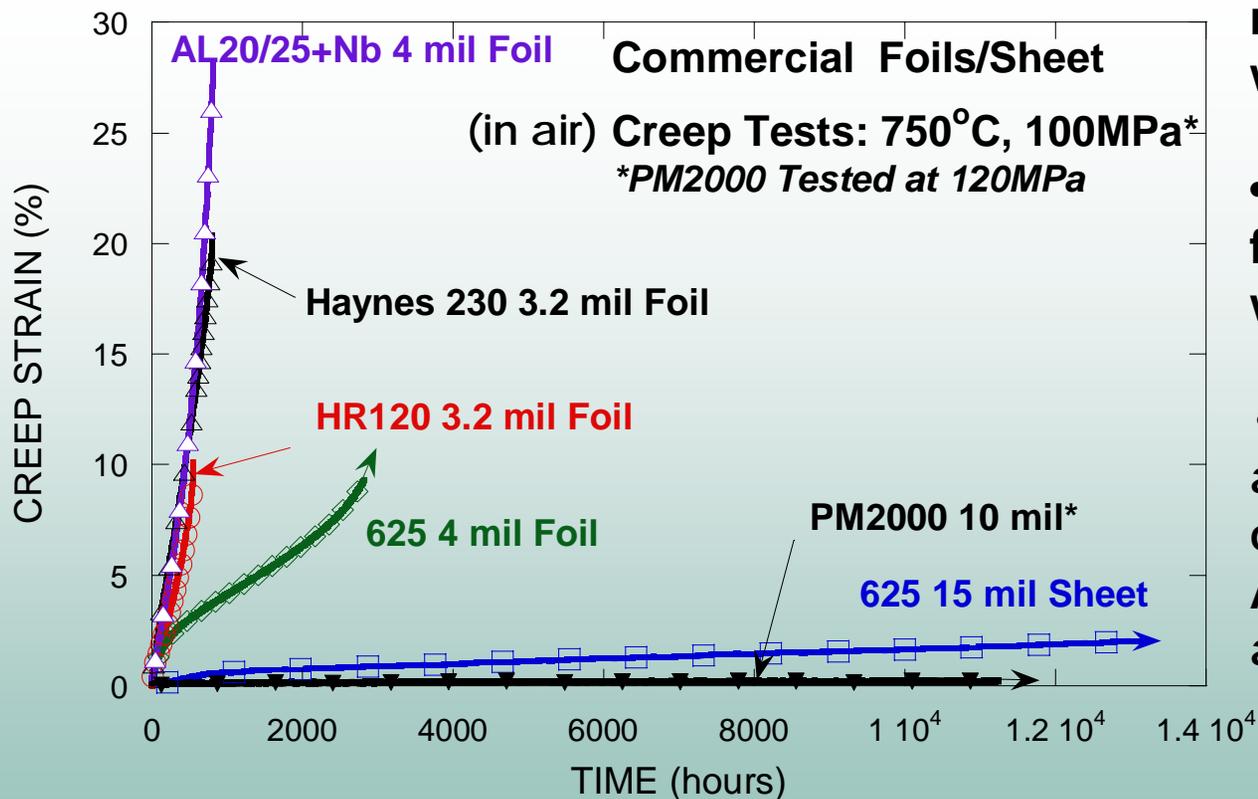
- AL 20/25+Nb
 - Fe-20Cr-25Ni-1.6Mo-1Mn-0.4Si-0.14Nb
- Haynes 120
 - Fe -25Cr-33Ni-1Mn-1Mo-0.05C-0.7Nb-0.2N
- PM2000
 - Fe-19Cr-5Al-0.4Ti-0.5Y₂O₃ (oxide-dispersed FeCrAl)
- Haynes 230
 - Ni-22Cr-14W-2Mo-3Fe-5Co-0.5Mn-0.4Si-0.3Al-0.1C-0.02La-.015B
- Alloy 625
 - Ni-21Cr-4.4Fe-9Mo-3.6Nb-0.02C-0.23Ti-0.16Al

**625 Supplied by ATI Allegheny Ludlum;
Other Sheet and Foils Supplied by Either Microturbine OEMs
or Foil Producers**

Creep Tests of Sheets and Foils at 704°C Have Been Performed



Creep Tests of Sheets and Foils at 750°C Have Been Performed

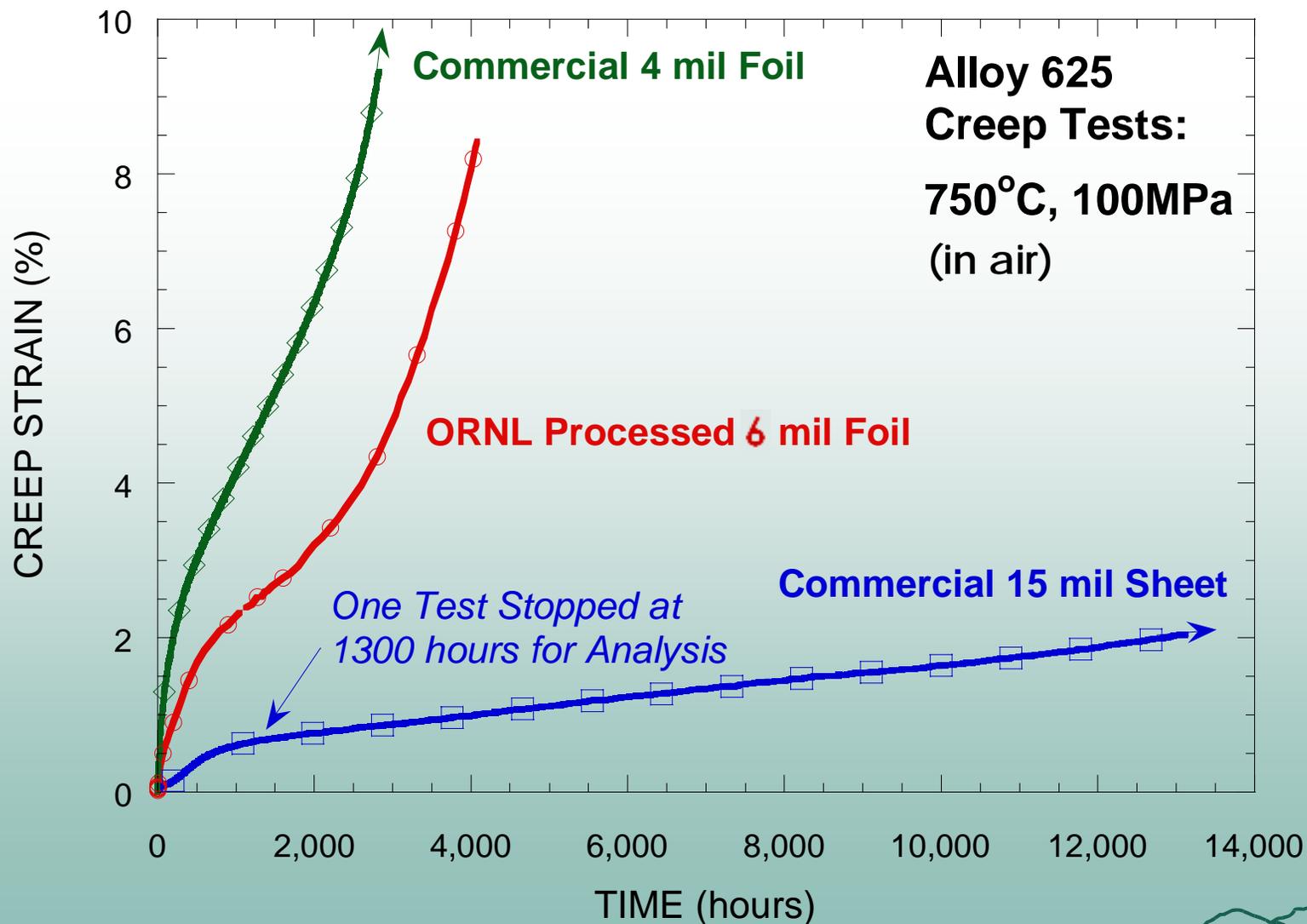


- Behavior of foil is not necessarily that of wrought product

- Material ranking for foils not same as wrought form

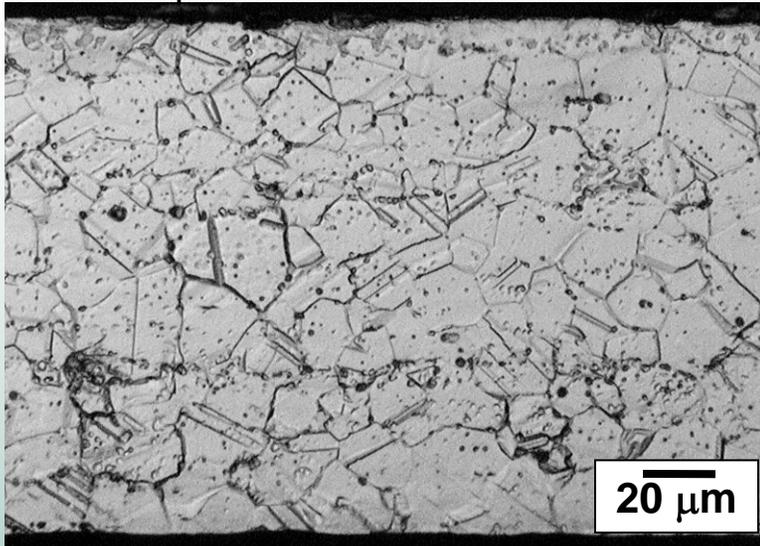
- For high temperature applications (e.g., fuel cells, heat exchangers), Alloy 625 may be most attractive.

Microstructures of Alloy 625 Sheets and Foils in Uncrept and Creep Condition (750°C, 100 MPa) Have Been Examined



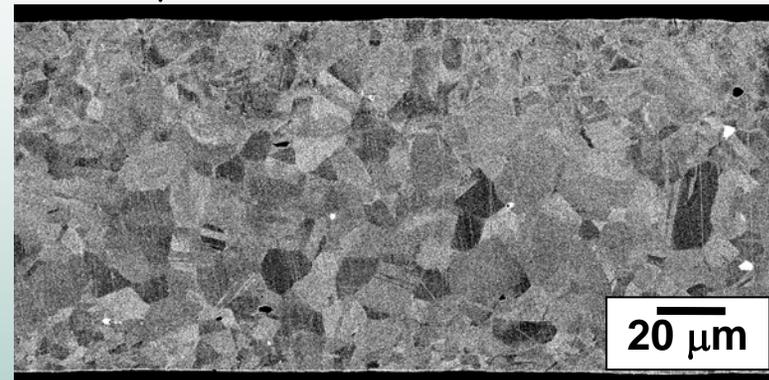
Average Grain Diameter of Alloy 625 Foils Estimated from \bar{l} , Mean Lineal Intercept Length

$$\bar{l} = 13.3 \mu\text{m}$$



ORNL Processed 6 mil (152 μm) foil
Optical micrograph,
uncrept foil in cross-section

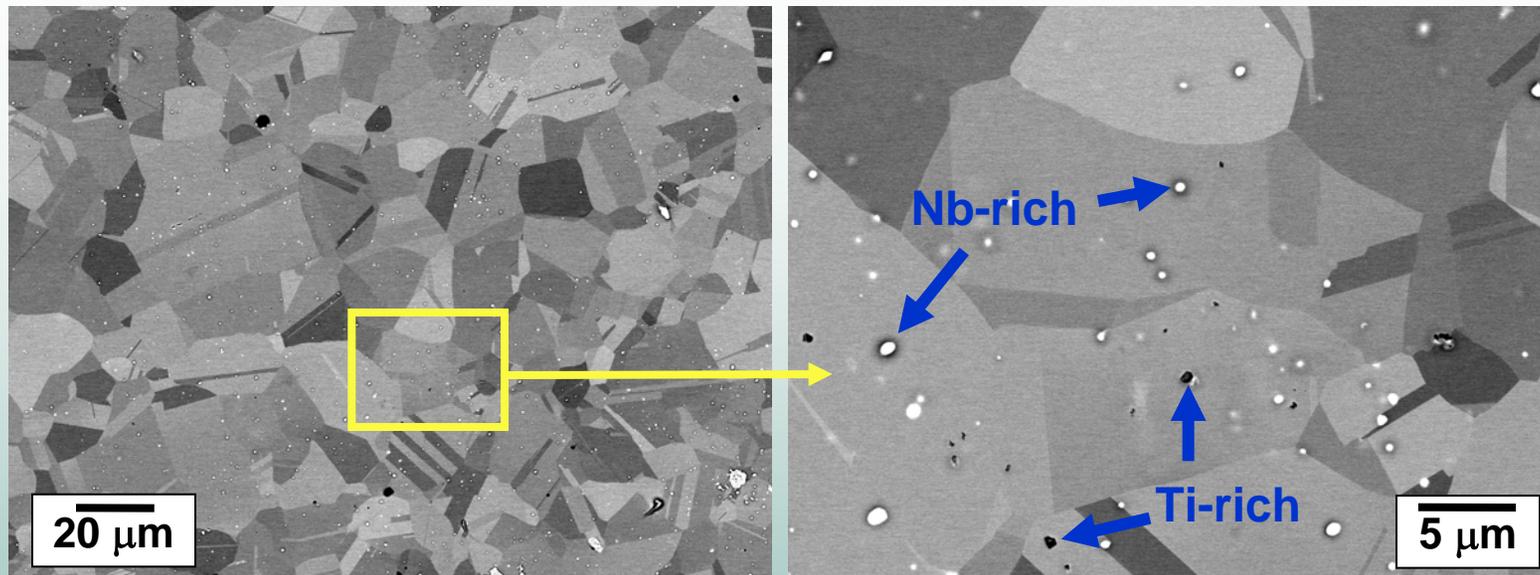
$$\bar{l} = 8.7 \mu\text{m}$$



AL supplied 4 mil (100 μm) foil
BSE SEM image,
uncrept foil in cross-section

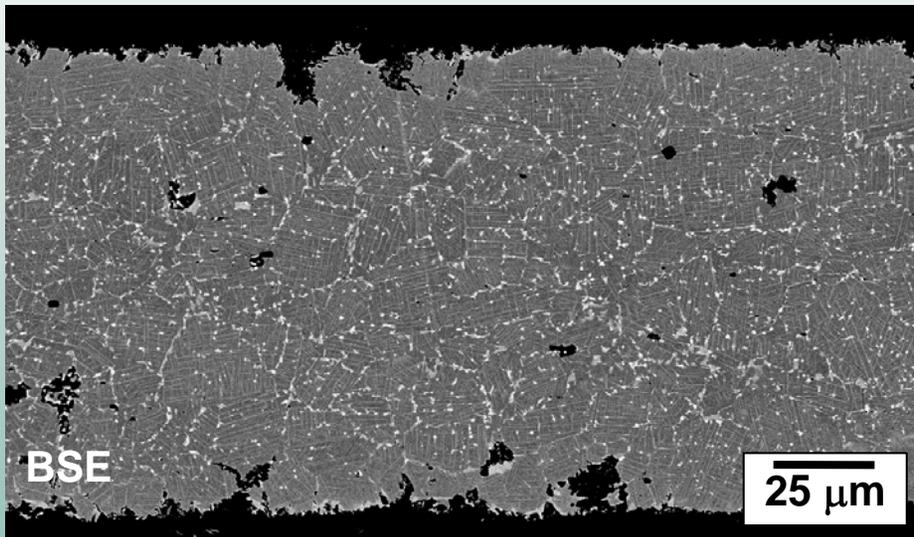
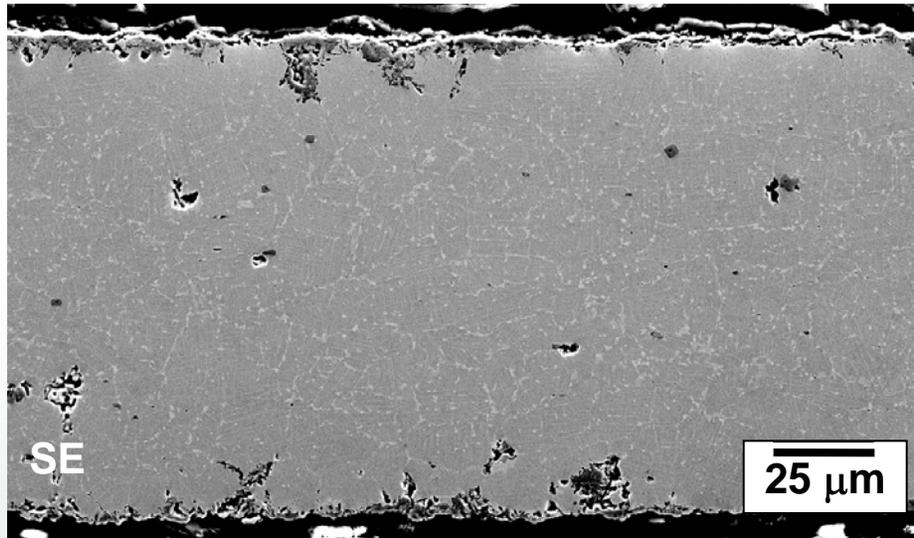
Uncrept 6 mil (152 μm) Alloy 625 Foils Have Clean Grain Boundaries, and Some Nb/Ti-Rich MX type Precipitates

ORNL processed foil



SEM BSE images, plan view of electropolished TEM Disks

**ORNL Processed 6 mil foil,
Crept 750°C, 100 MPa, 4510 h**



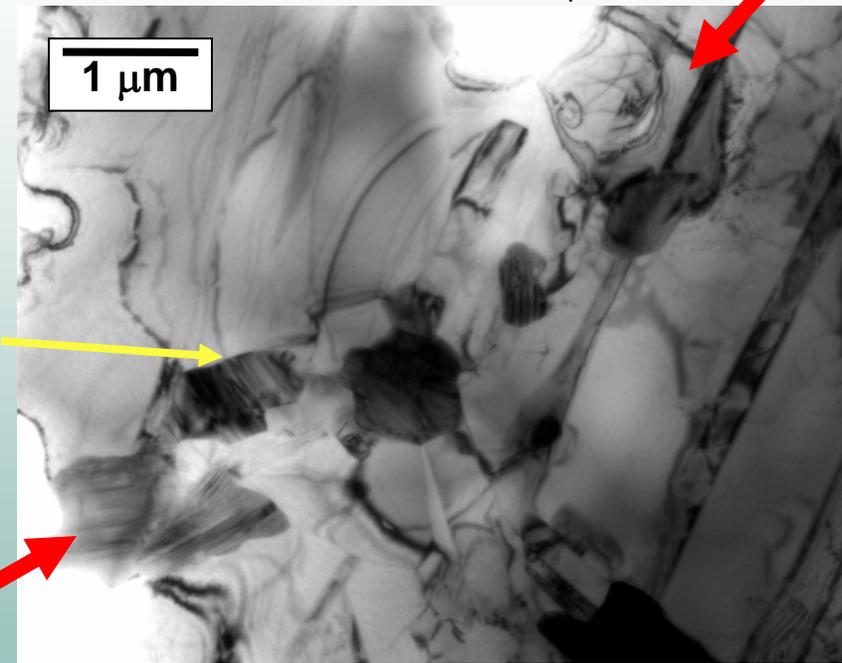
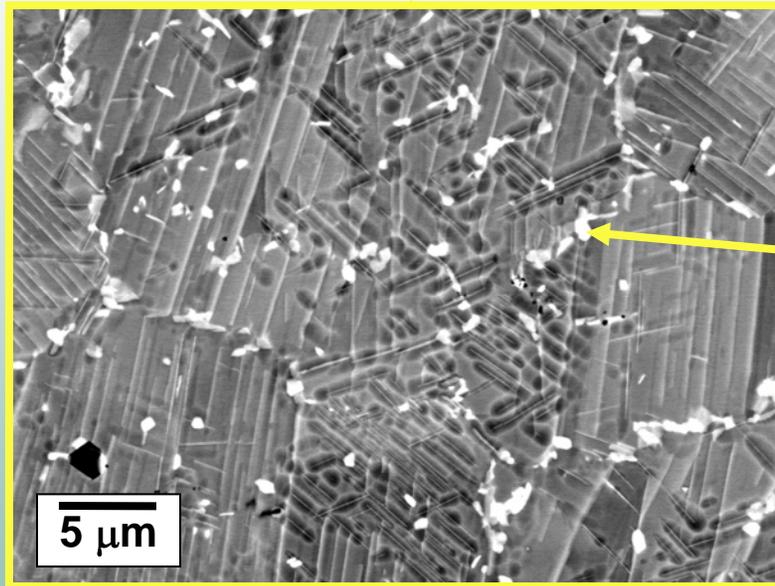
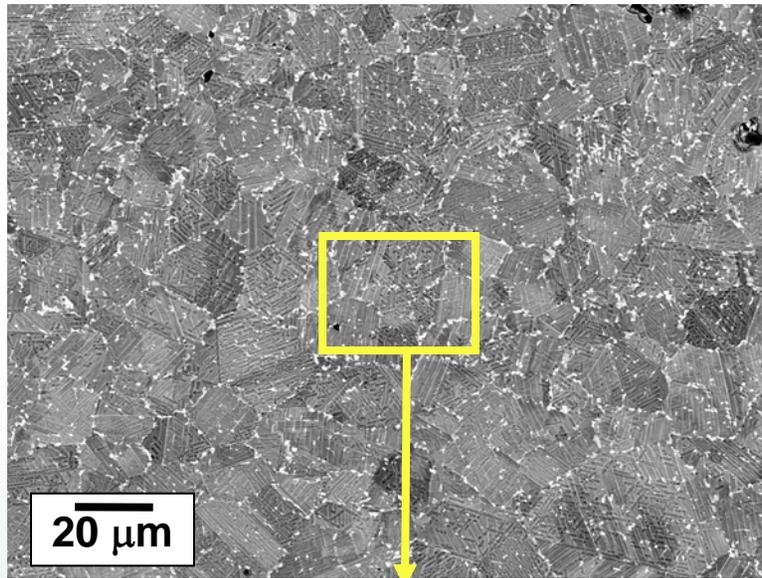
SEM Images, Same Field-of-View

Significant Changes in Microstructure Occur During Creep-testing 0.006" Alloy 625

- Oxide develops on surface, with deepest penetration into foil along grain boundaries
- Precipitate formation in grain boundaries

During Creep Testing, Alloy 625 Develops Grain Boundary Precipitates and a Phase, Having a Plate Morphology, Within Grains

ORNL Processed 6 mil foil
crept 750°C, 100 MPa $t_r=4510$ h



SEM BSE images from
electropolished TEM Disk

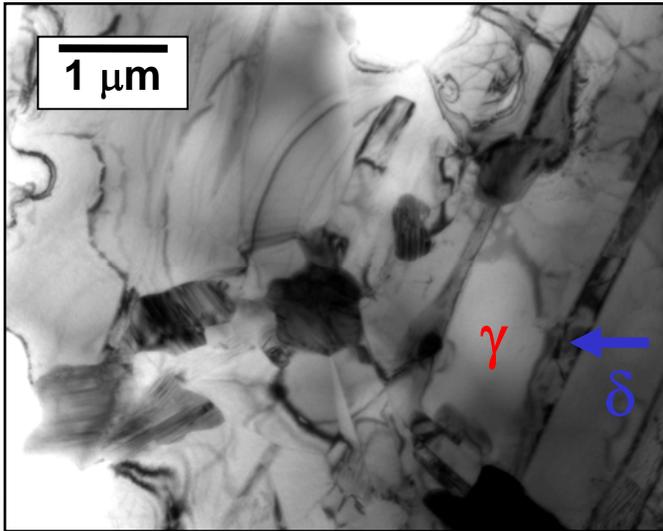
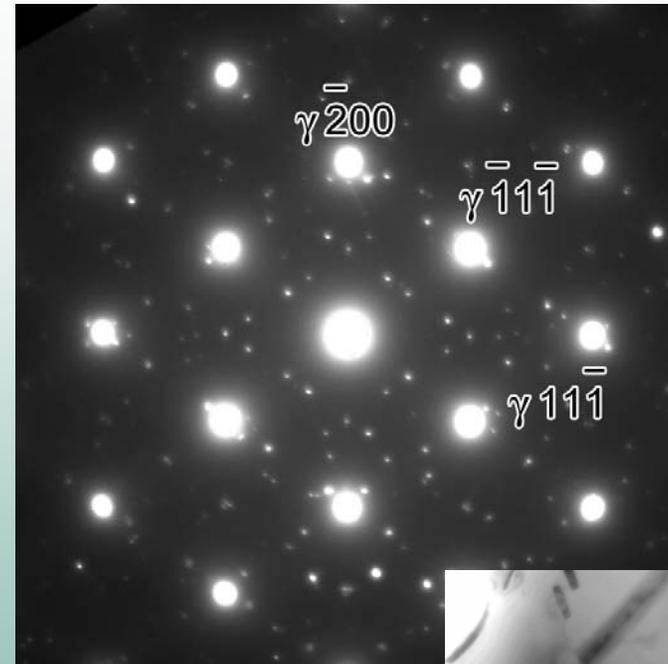
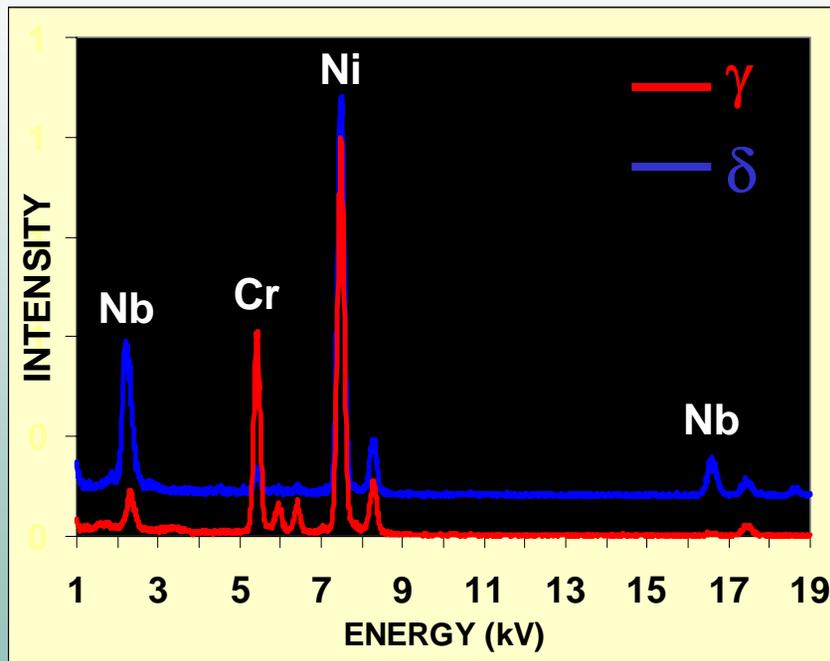


Plate Phase in Crept Alloy 625 Is Identified as Equilibrium Orthorhombic δ (Ni_3Nb)

Alloy 625, crept 750°C, 100 MPa $t_c=4510$ h



$B = [011]_{\gamma}$



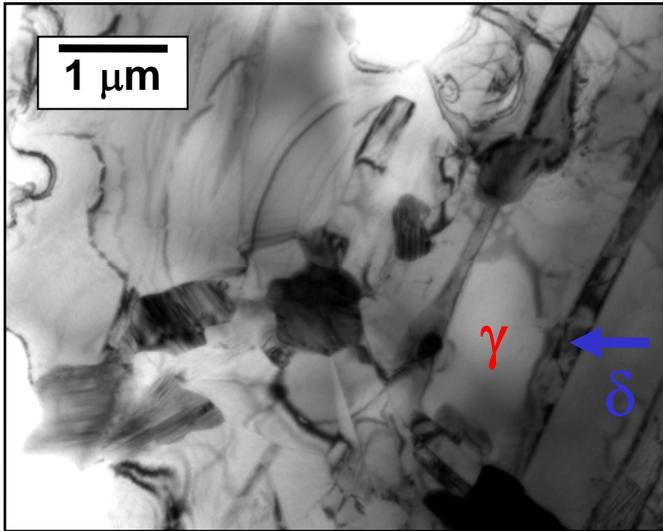
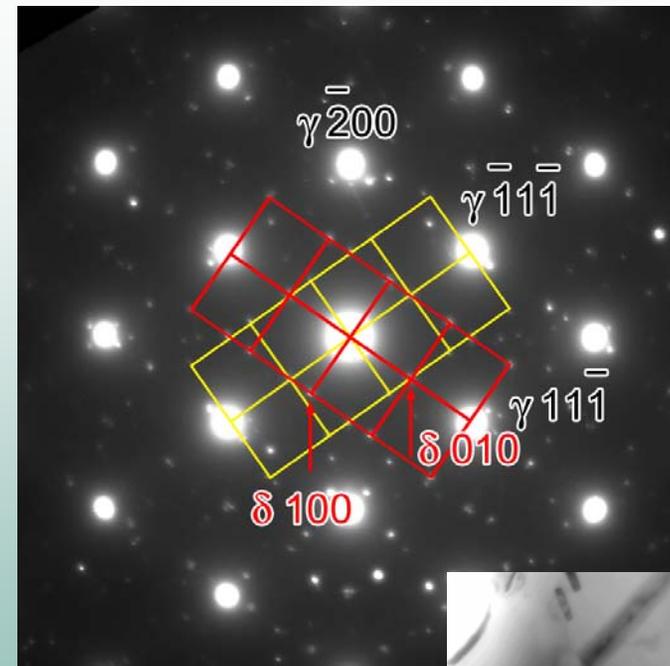
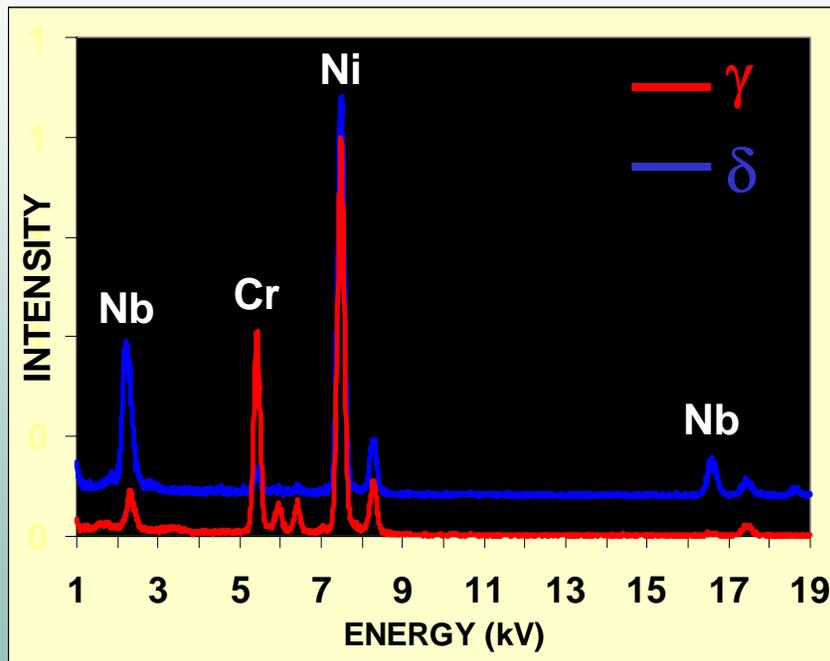


Plate Phase in Crept Alloy 625 Is Identified as Equilibrium Orthorhombic δ (Ni_3Nb)

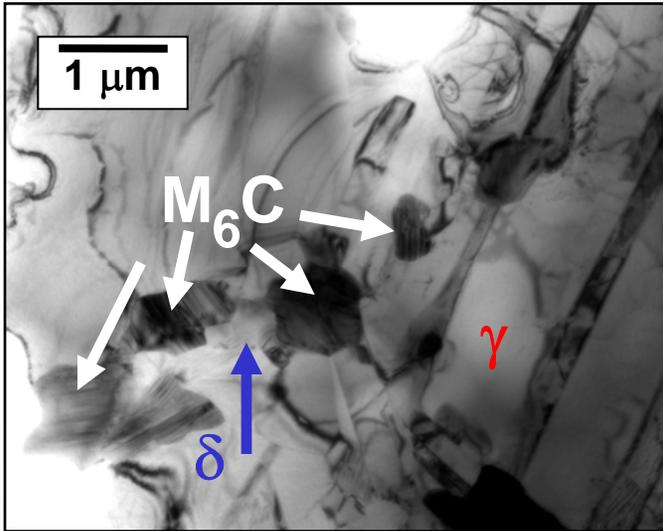
$$[011] \gamma \parallel [100] \delta$$

$$(11\bar{1}) \gamma \parallel (010) \delta$$



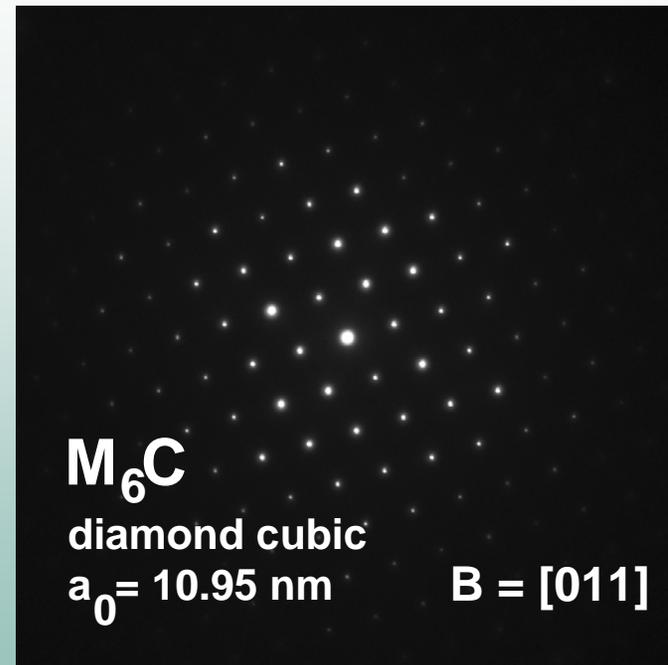
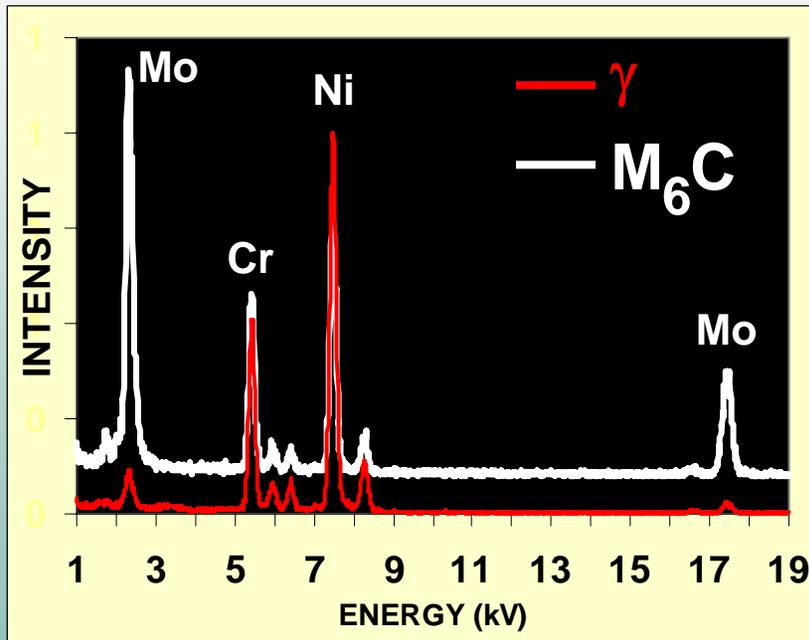
(2 rotational
variants)



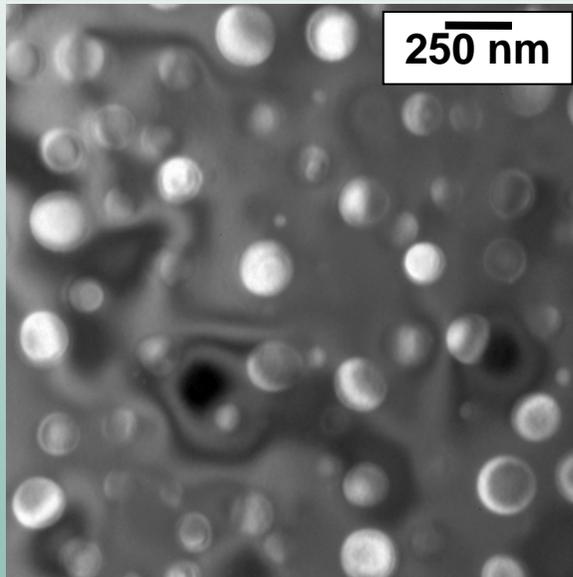
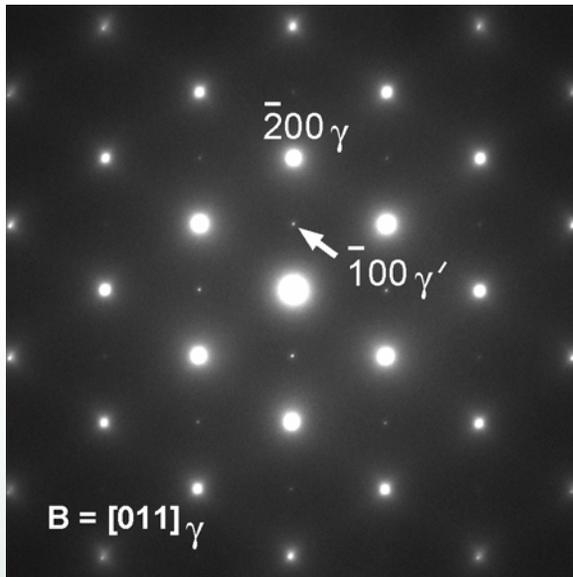


crept 750°C, 100 MPa

Grain Boundaries in Crept 6 mil Alloy 625 are Stabilized with M_6C Precipitates (some δ also)

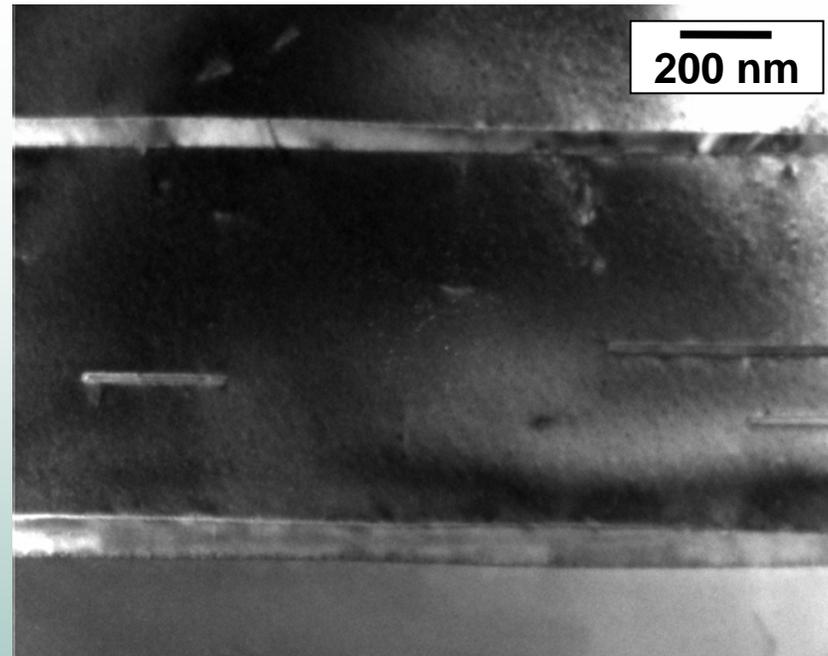


**Service-Aged Pyromet 31V
(Ni-22Cr-15Fe)**

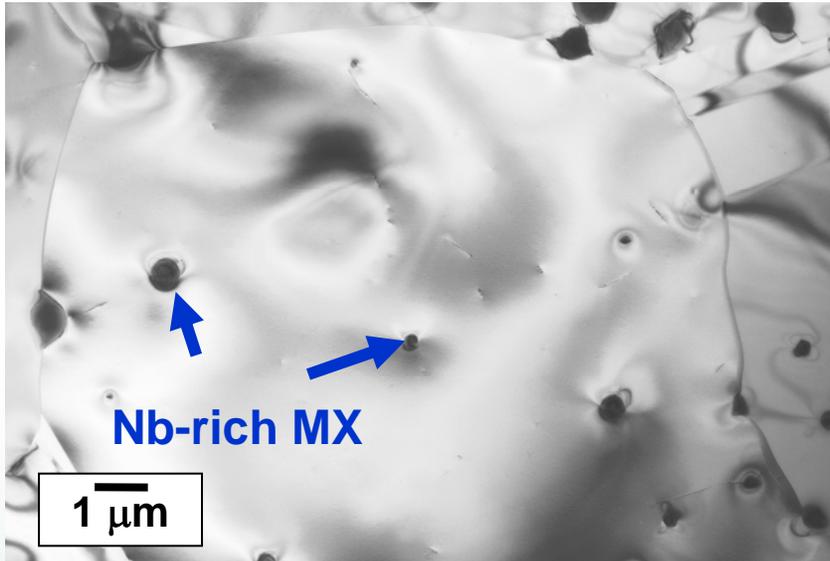


**Centered Dark Field Imaging in
TEM Can Reveal γ' Within γ**

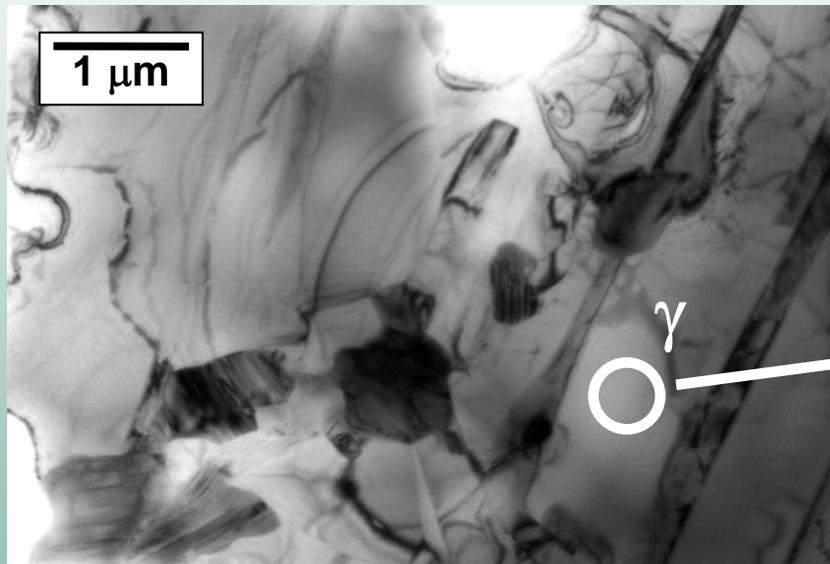
**TEM Dark Field Image, Centered
Midway Between (000) and $(\bar{2}00)_\gamma$**



**ORNL Processed 6 mil foil,
Crept 750°C, 100 MPa, 4510 h**



uncrept



crept 750°C, 100 MPa

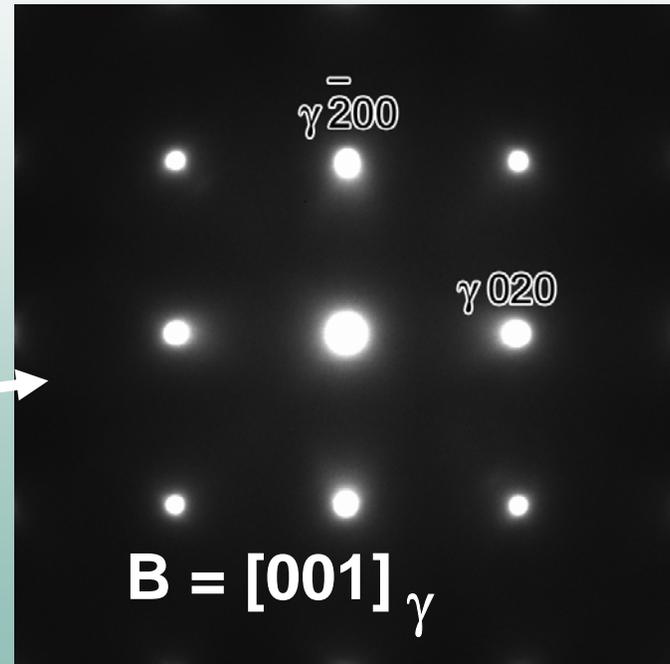
During Creep Testing At 750°C Alloy 625 Foil Does Not Develop a Finely Dispersed Precipitate Phase

MX consumed by δ laths

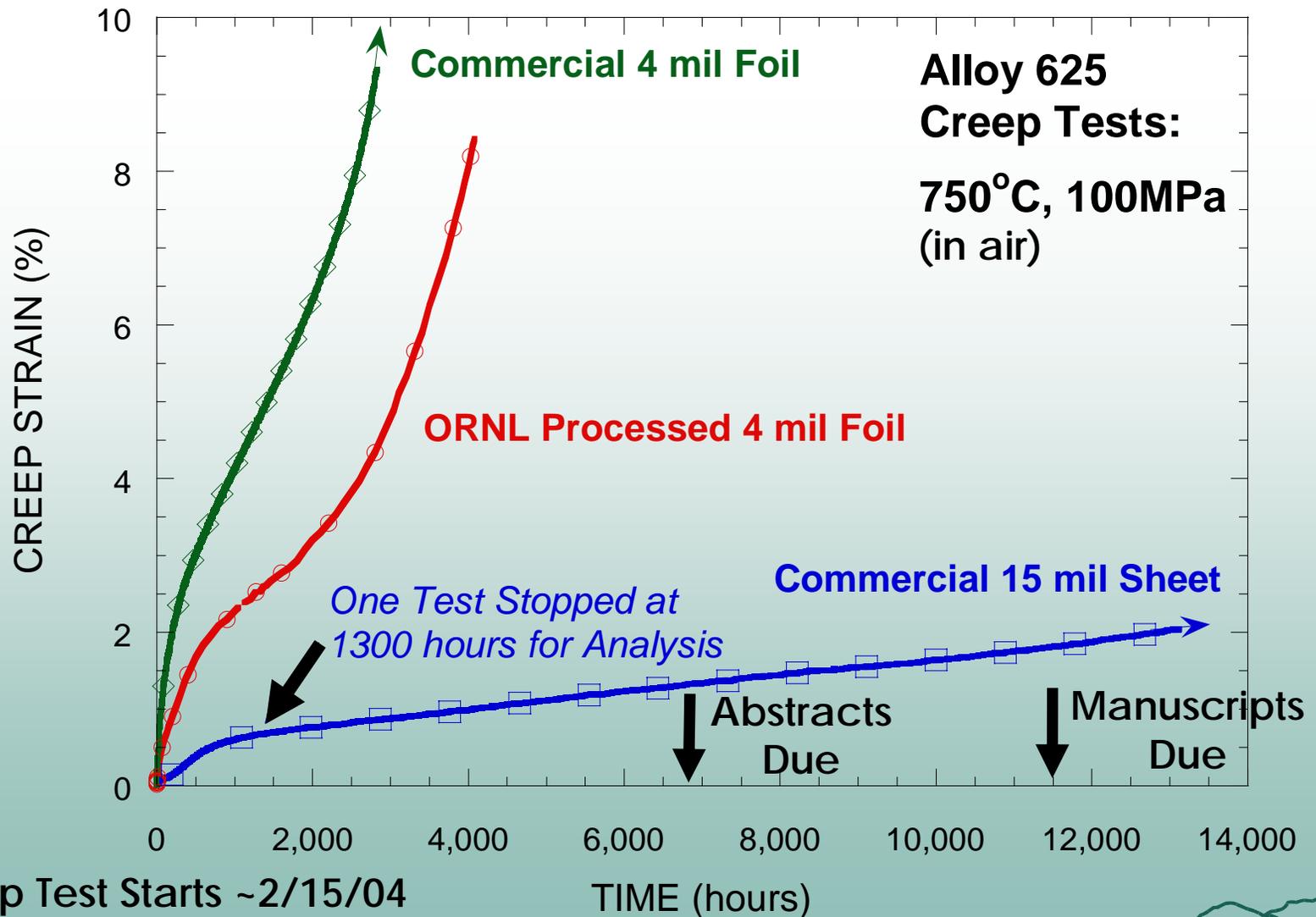
No γ' $\text{Ni}_3(\text{Al}, \text{Ti})$

No γ'' (metastable Ni_3Nb)

**No topologically close-packed
phases (e.g. μ , σ) observed**

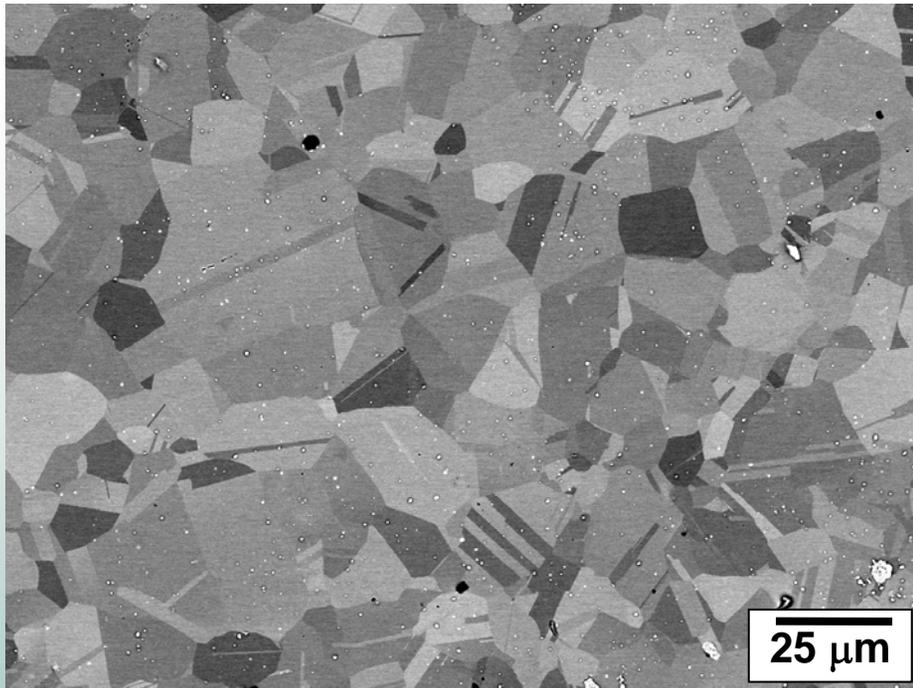


Microstructure of Alloy 625 Foils in Both Uncrept and Crept Condition (750°C, 100 MPa) Have Been Examined



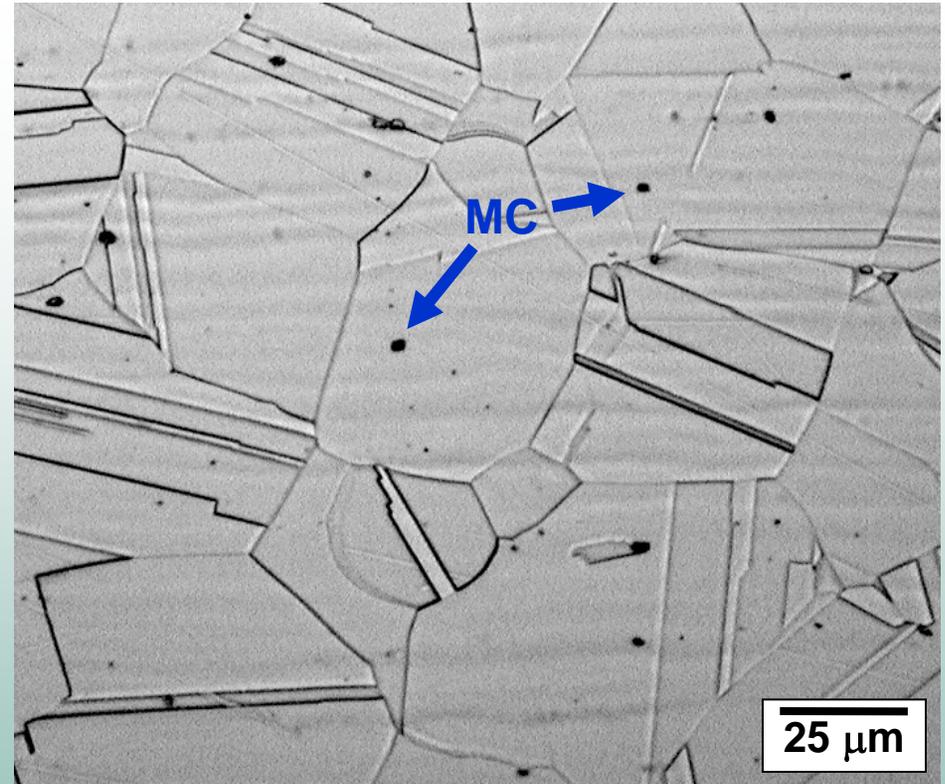
Other Than Having Significantly Larger Grain Size, As-Processed 15 mil Alloy 625 Sheet Is Similar to 6 mil Foil

$$\bar{L} = 13.3 \mu\text{m}$$



ORNL Processed 6 mil (100 μm) foil
BSE SEM Image,
uncrept foil, plan view

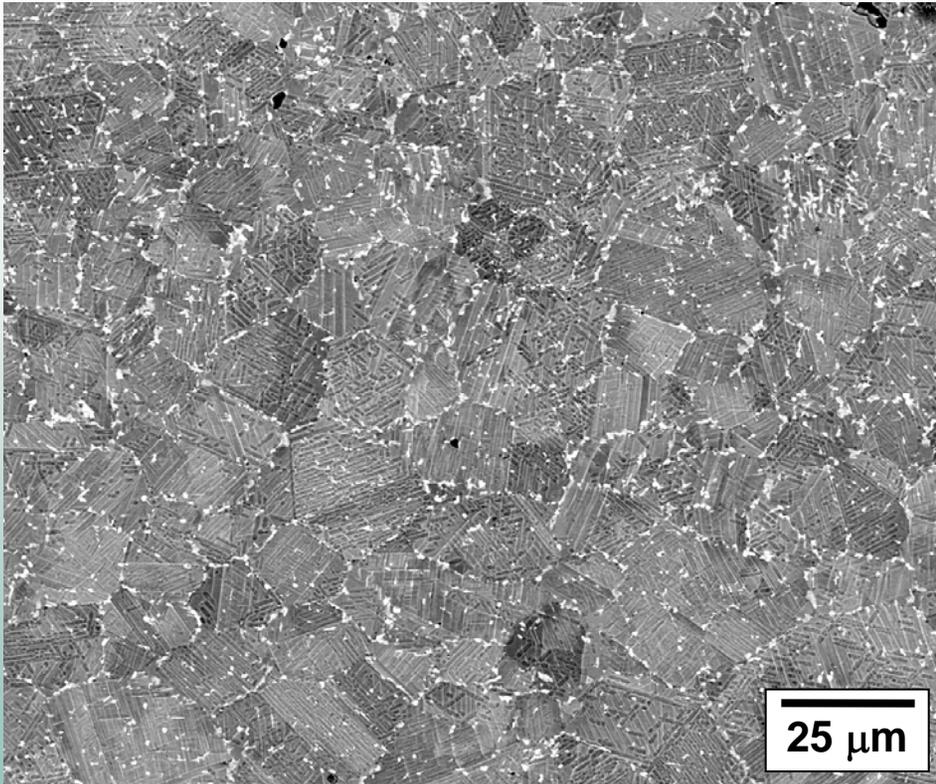
$$\bar{L} = 38.7 \mu\text{m}$$



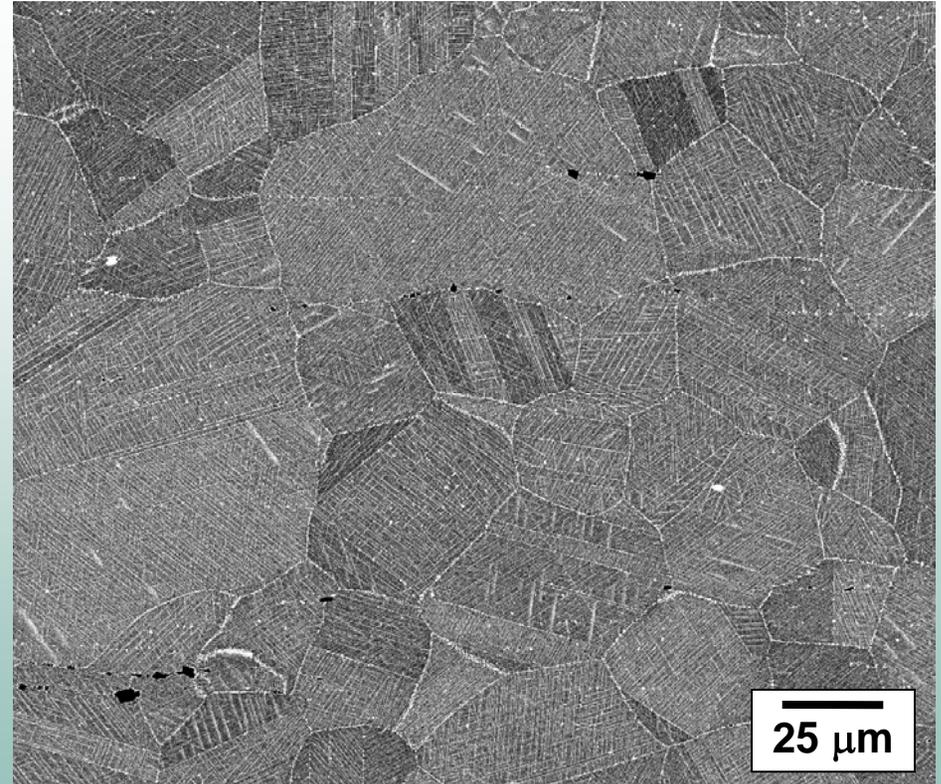
15 mil (375 μm) foil Optical
micrograph,
uncrept foil, cross-section view

15 mil Alloy 625 Sheet, Creep-Tested to Reach Steady-State, Develops Morphology Similar to Creep-Tested 6 mil Foil

ORNL Processed 6 mil foil
crept 750°C, 100 MPa $t_r=4510$ h

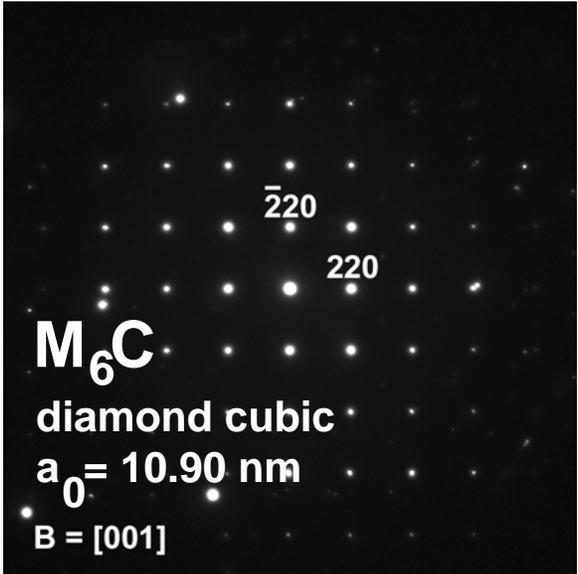


15 mil Sheet, Crept 750°C, 100 MPa
1300 h (Interrupted)

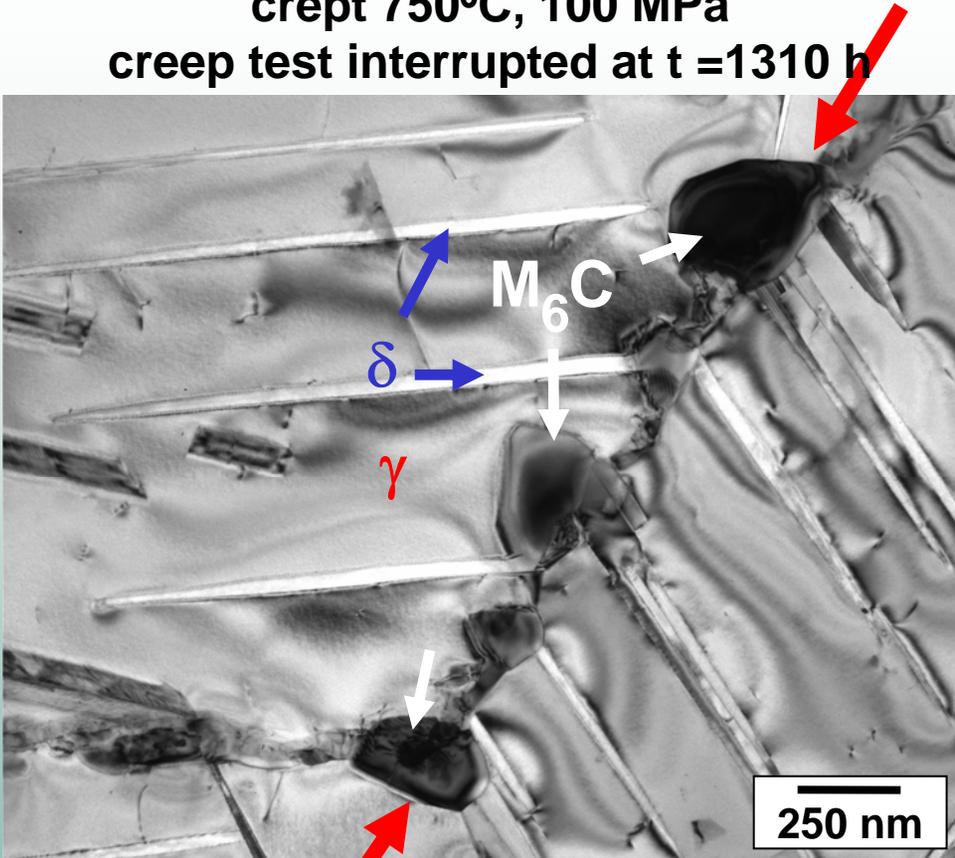
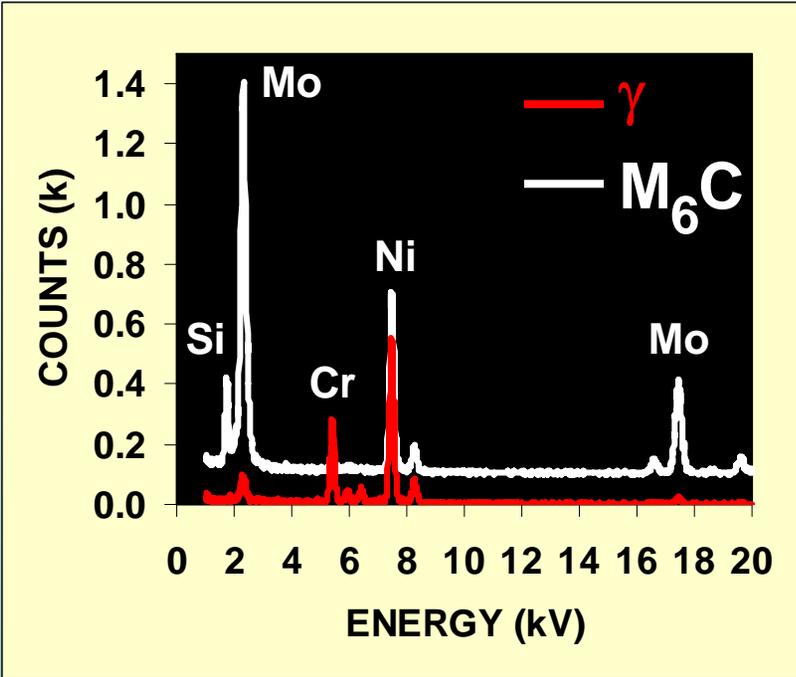


BSE SEM Images

Grain Boundaries in Crept 15 mil Alloy 625 are Stabilized with M_6C Precipitates (some δ also)



15 mil (375 μm) foil
crept 750°C, 100 MPa
creep test interrupted at $t = 1310 \text{ h}$



Summary

- Foils and sheet of Alloy 625, considered for use in recuperators, have been investigated
 - Creep behavior of foil is not that of the sheet
 - (available creep data for candidate materials in sheet form may not be extended to same material as a foil)
 - During creep testing at 750°C, 100MPa
 - Precipitation of M_6C and δ phases, which stabilize grain boundaries during creep
 - No precipitation of finely dispersed phases within austenite
 - No embrittling topologically close-pack phases (e.g. σ , μ , Laves)
 - Difference in creep behavior due differences in grain size
 - Additional processing to obtain foil resulted in reduction in grain size, thereby increasing potential for grain boundary sliding and creep rate