Redistribution of Carbide Precipitates during Creep of Alloy 617

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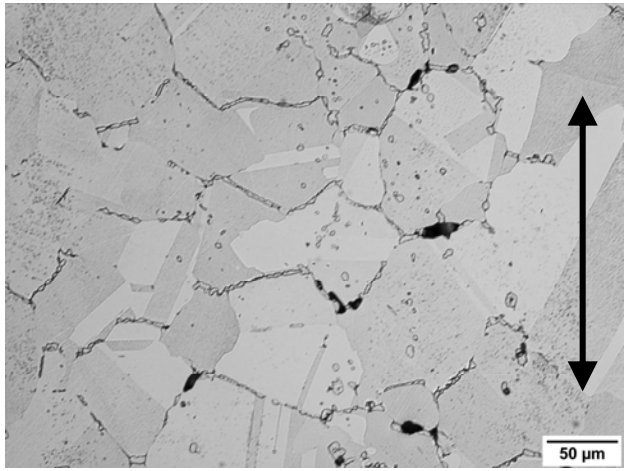

Funded by Idaho National Laboratory LDRD

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Properties of alloy 617

- Solid solution strengthened nickel-based superalloy

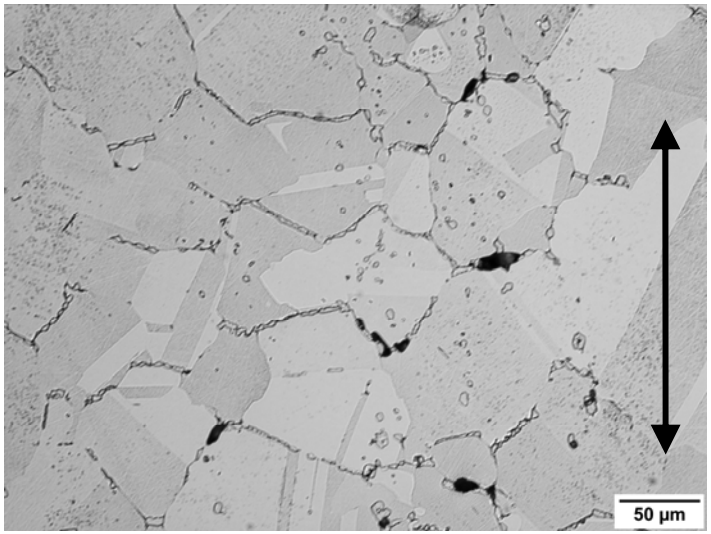
solution strength:	oxidation resistance:
Mo and Co	**Cr and Al**

- Elevated temperature strength from precipitation of carbides
- Major precipitate phases are chromium-rich ($M_{23}C_6$) and molybdenum-rich (M_6C) carbides
  - Cr-rich carbides reside primarily on the grain boundaries
  - Mo-rich carbides are usually found within the grains

S. Kihara, A. Ohtomo, Y. Saiga, and J. B. Newkirk, *Metallurgical Transactions* (1980) **11A**, 1019-31.
Y. S. Lim, J. S. Kim, H. P. Kim, and H. D. Cho, *Journal of Nuclear Materials* (2004), **335**, 108-114.
H. Liu, M. Gao, D. G. Harlow, and R. P. Wei, *Scripta Metallurgica et Materialia* (1995), **32**,1807-1812.

Precipitate redistribution

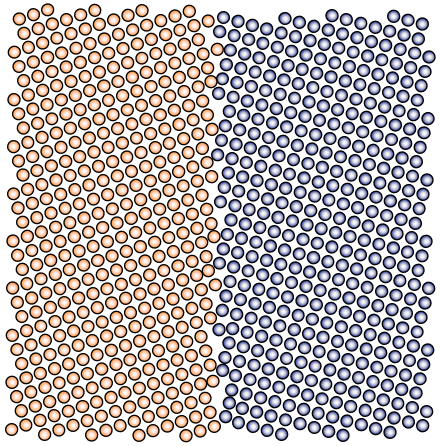
- During creep, intragranular carbides are believed to re-dissolve and re-precipitate as grain boundary carbides
- Carbides have been shown to redistribute to grain boundaries in tension during creep
- Difference in stability of the carbides in compressive versus tension is proposed as the driving force for the re-distribution
- After creep tests, the majority of the grain boundary precipitates were shown to be Cr-rich ($M_{23}C_6$) carbides due to the higher diffusivity of chromium versus molybdenum in nickel-based alloys
- Mo-rich carbides tended to be more resistant to dissolution



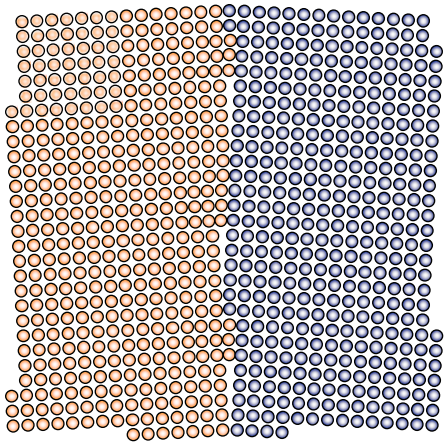
S. Kihara, A. Ohtomo, Y. Saiga, and J. B. Newkirk, *Metallurgical Transactions* (1980) **11A**, 1019-31.
L. Z. He, Q. Zheng, X. F. Sun, G. C. Hou, H. R. Guan, and Z. Q. Hu, *Journal of Materials Science* (2005), **40**, 2959-64.
G. D. Smith and D. H. Yates, Orlando, FL, USA, 1991.

Role of grain boundaries in 617

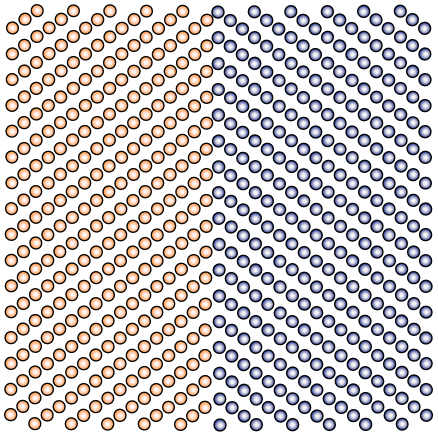
- Grain boundary character influences carbide precipitation
- Preferential precipitation is driven by differences in grain boundary energy



General boundaries: carbides are coarse and irregular



LA & CSL boundaries: carbides are fine and densely distributed



Coherent twins ($\Sigma 3$): no carbides

Y. S. Lim, J. S. Kim, H. P. Kim, and H. D. Cho, *Journal of Nuclear Materials* (2005), **335**, 108-114.
H. Liu, M. Gao, D. G. Harlow, and R. P. Wei, *Scripta Metallurgica et Materialia* (1995), **32**, 1807-1812.

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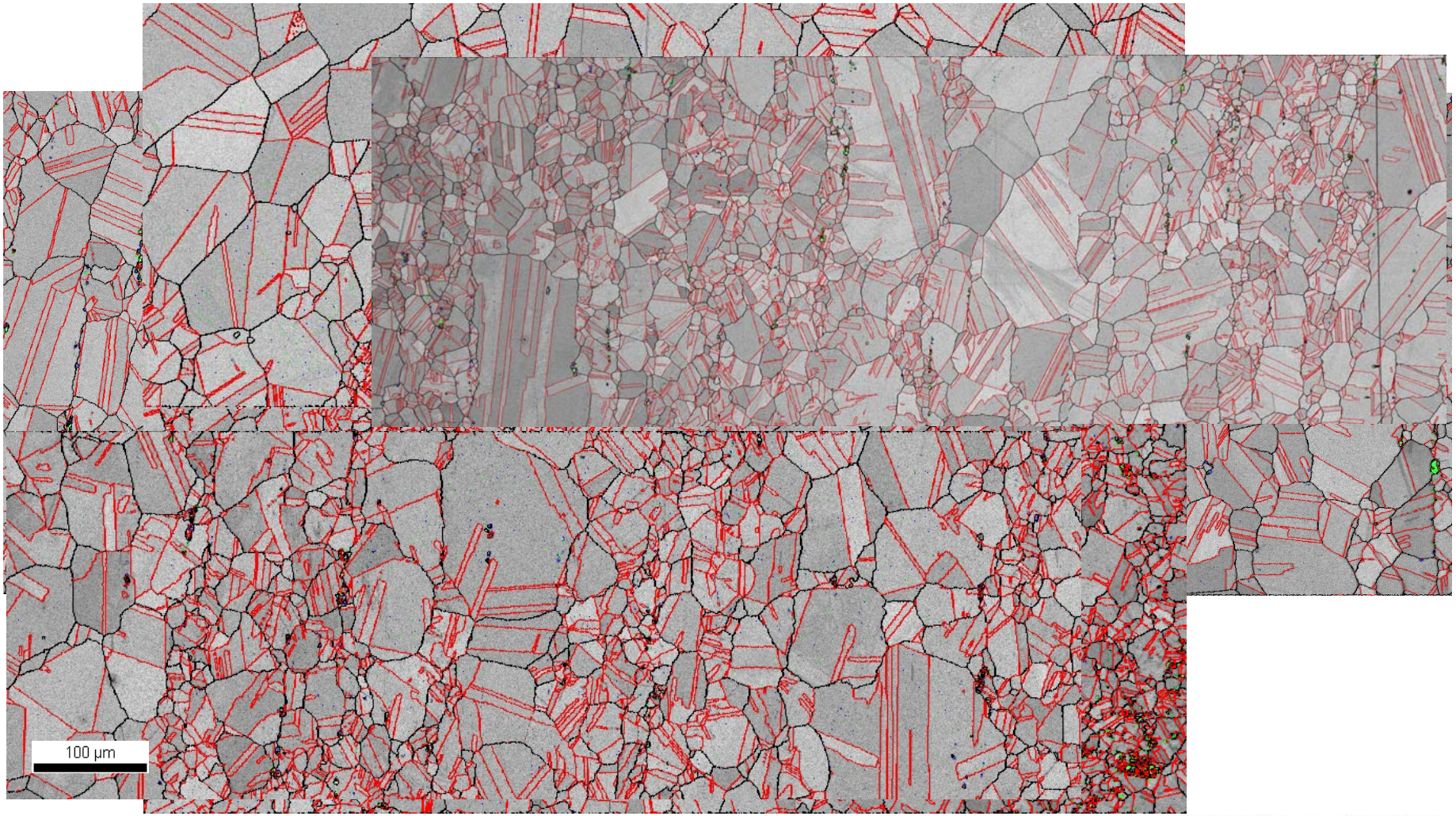
As-Received 617



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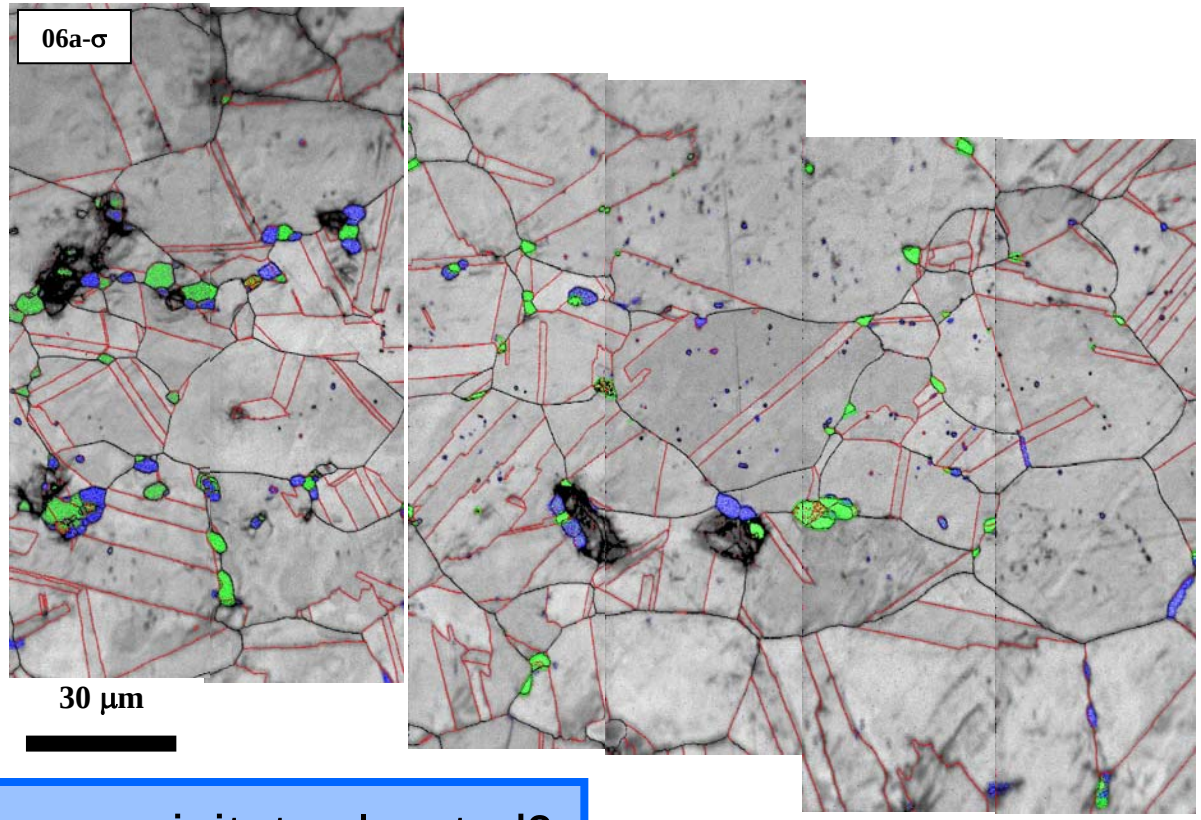
Creep testing at INL

Specimen ID	Creep Temp (°C)	Creep Stress (MPa)	Creep Time (hrs)	Total Creep Stain (%)
02	1000	24.7	350	14.3
06	1000	25	137	14.2
06a	1000	18.6	497	13.9
08	900	40.25	528	17.1
09	900	50.48	265	14.2
10	900	35	265	8.2

Quantifying precipitate distribution



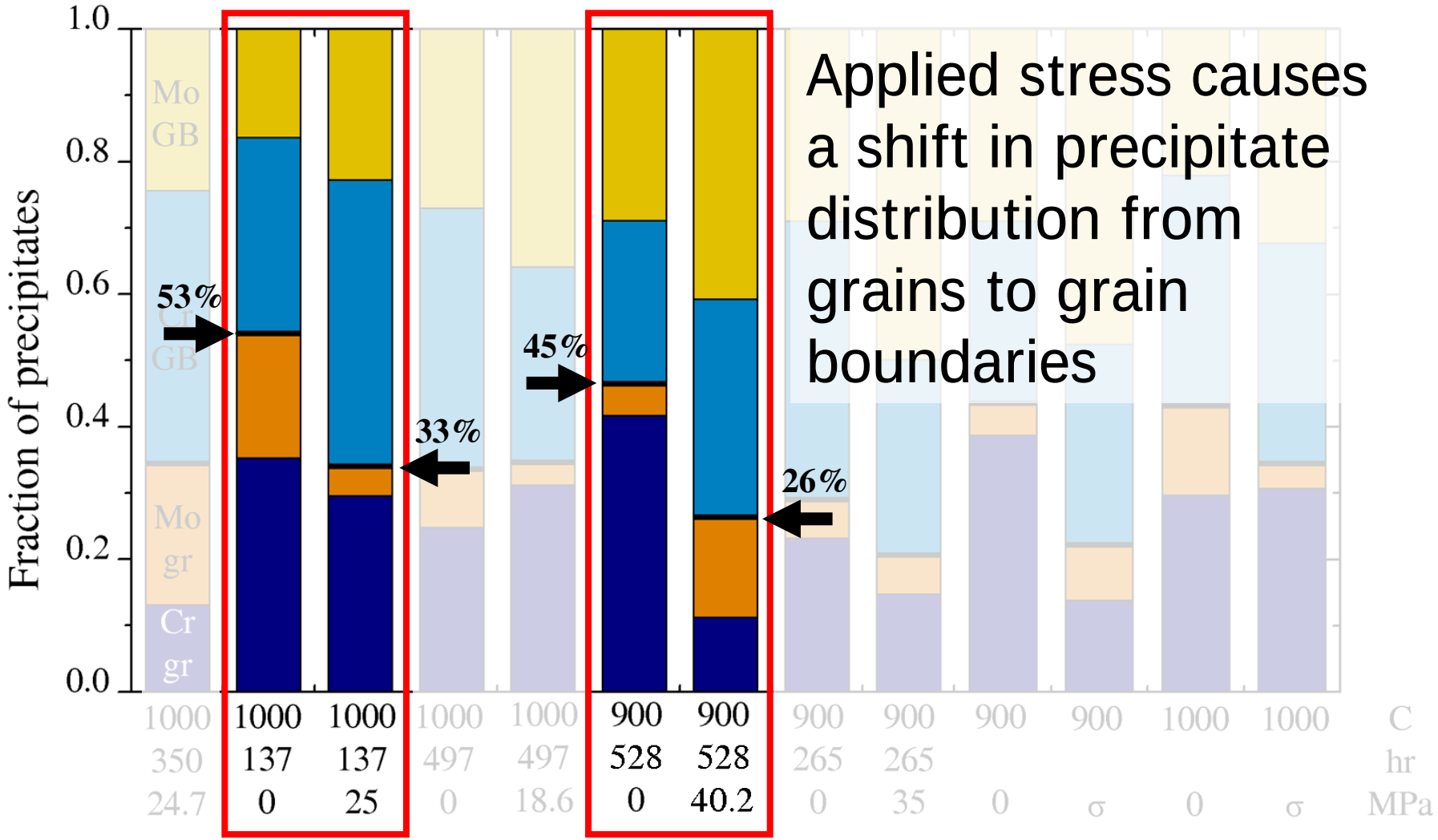
Where are precipitates located?

- In grain interiors?
- On grain boundaries?
- At GB triple junctions?

Does precipitate type matter?

- Mo-rich
- Cr-rich

Location of precipitates (grain or GB)



Applied stress causes a shift in precipitate distribution from grains to grain boundaries

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Effect of boundary inclination

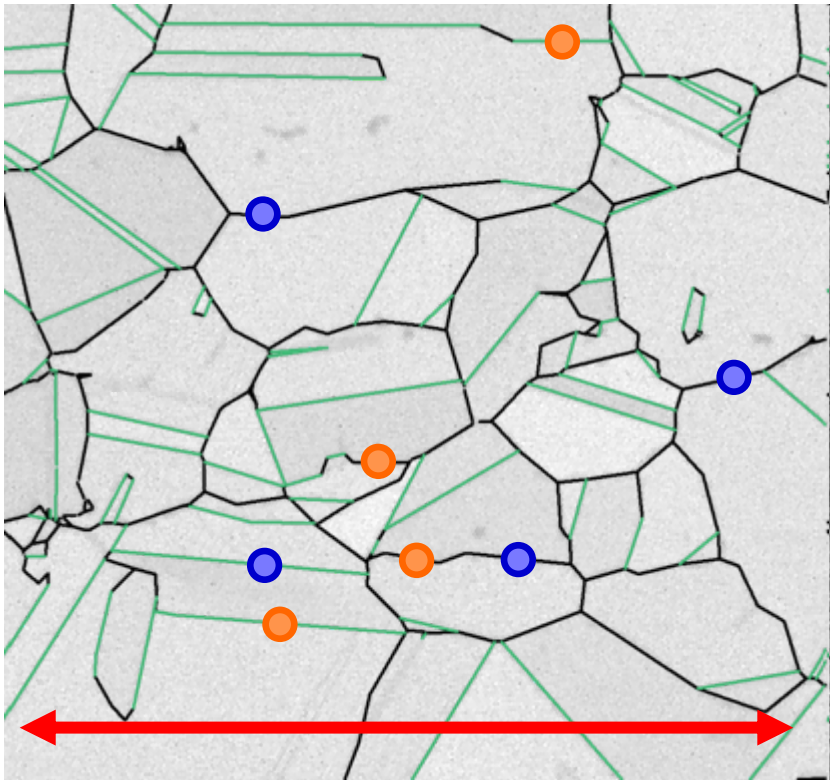
$$f(i,\theta) = \frac{B_{i,\theta}}{N_\theta}$$

$B_{i,\theta}$: # of boundaries with angle θ and ppt type i

N_θ: # of boundaries with angle θ

i: ppt type (none, Cr-rich, Mo-rich, both)

θ: inclination angle (90° is $\perp$ to σ-axis)



Boundaries between 0-10°:

14 boundaries w/ no ppts

3 boundaries w/ Mo-rich ppts

1 boundary w/ both ppts

3 boundaries w/ Cr-rich ppts

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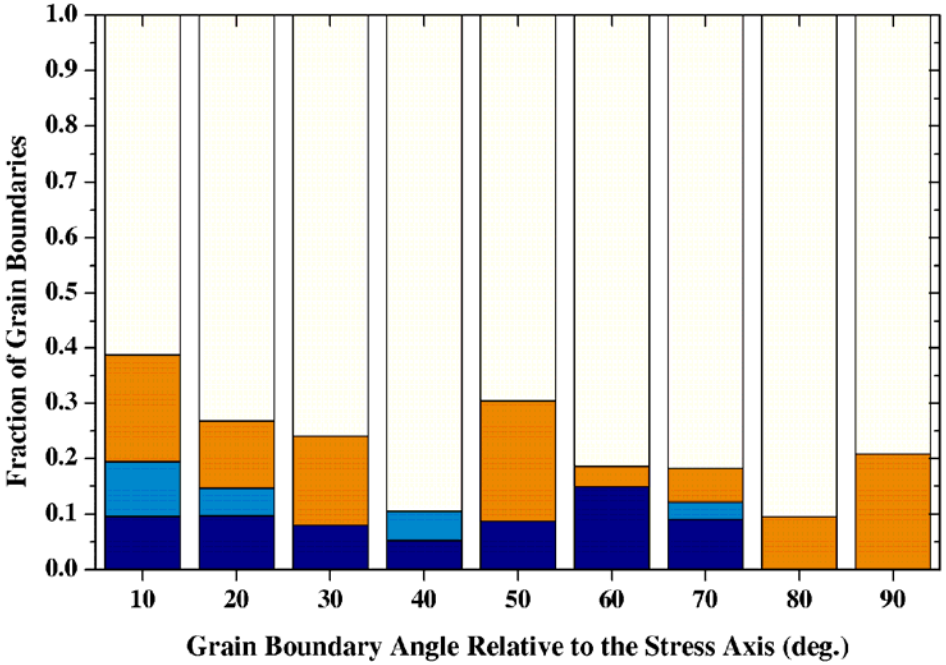
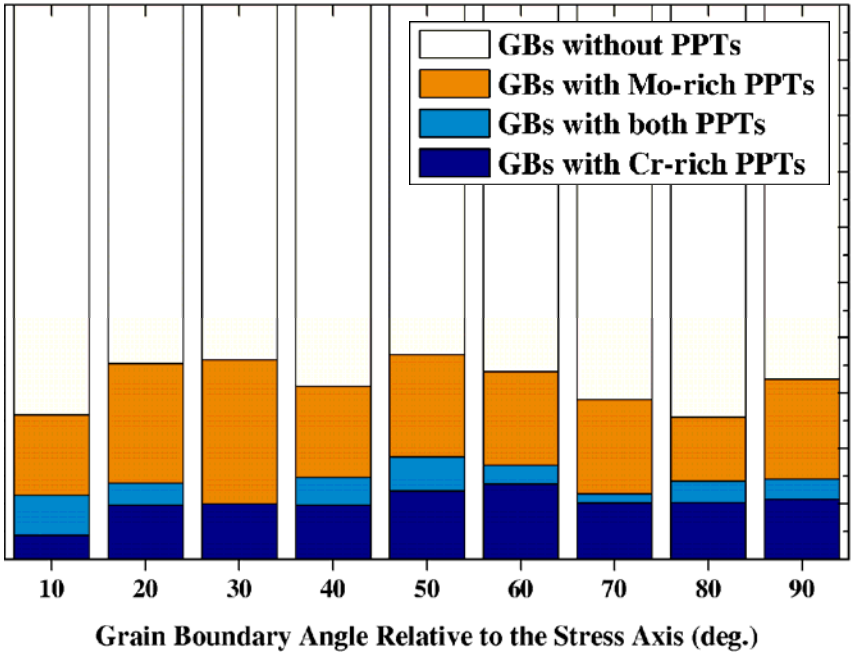
Effect of boundary inclination

T = 1000°C
t = 497 h

No applied stress
(grip end)



Applied stress
(gauge section)



expected shift

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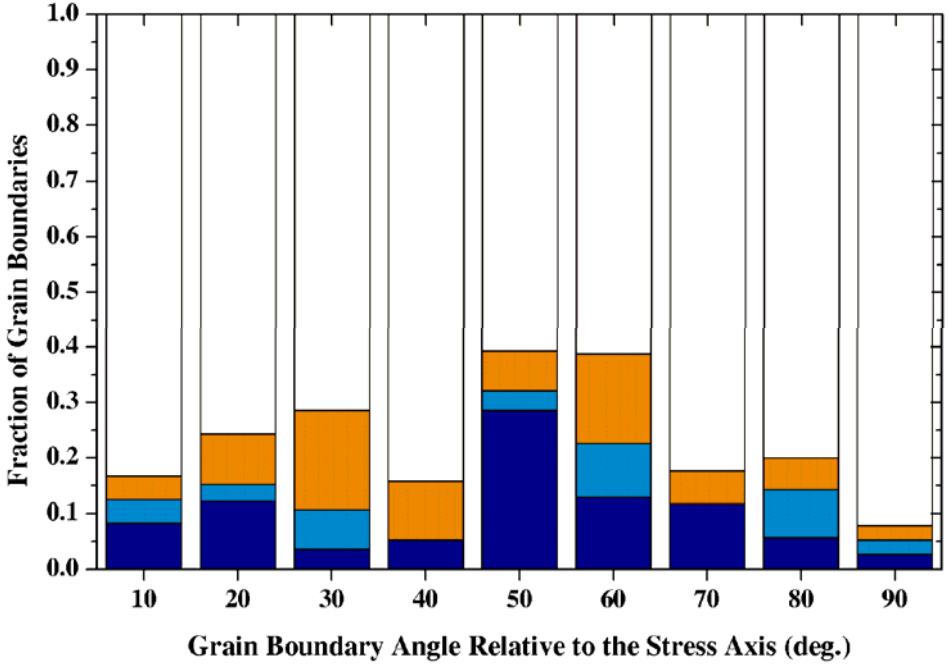
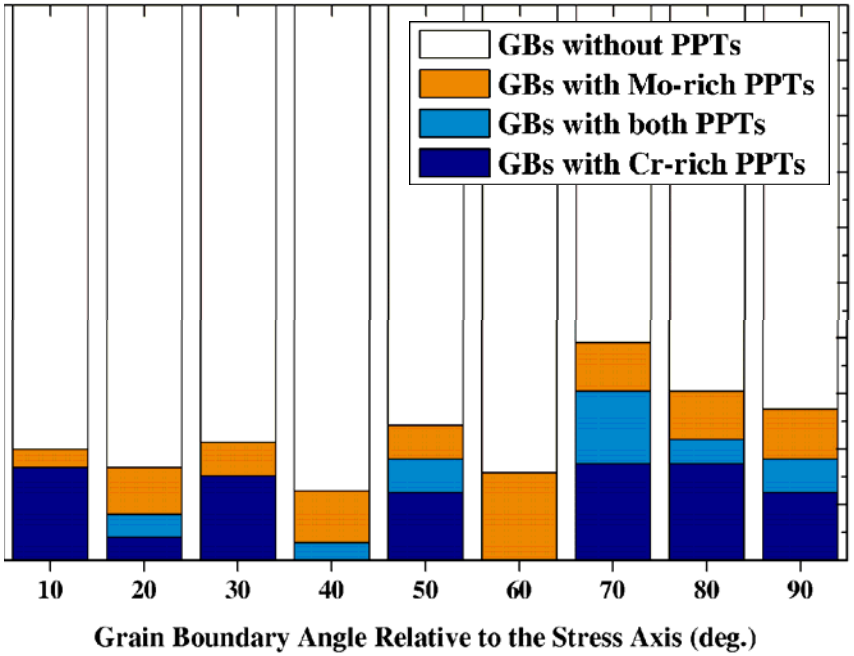
Effect of boundary inclination

T = 1000°C
t = 137 h

No applied stress
(grip end)



Applied stress
(gauge section)



expected shift

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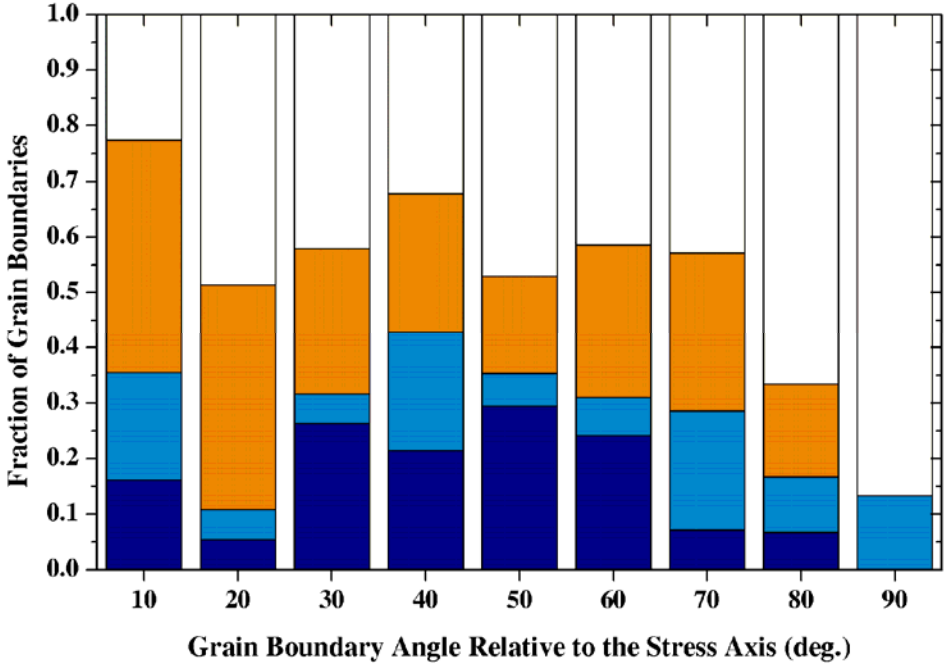
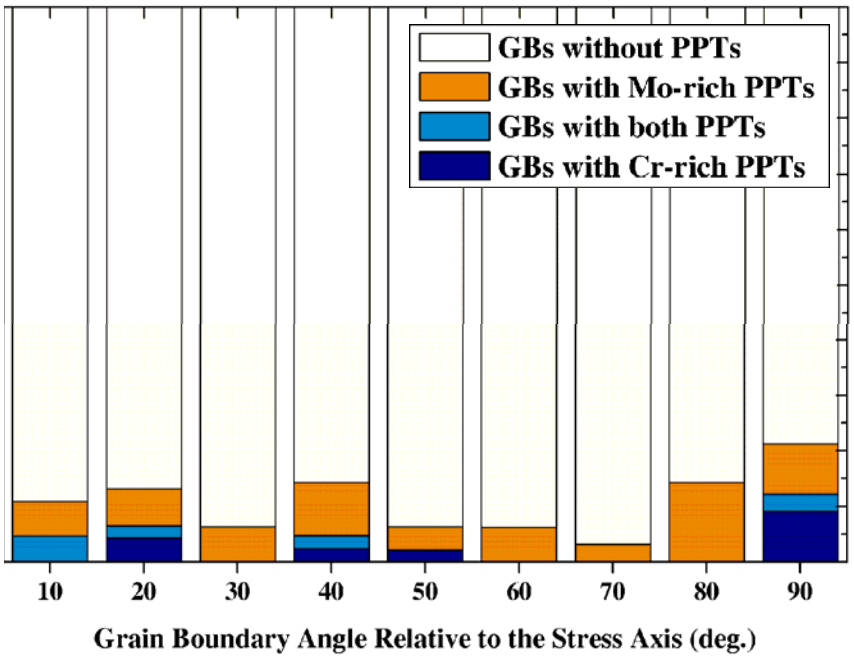
Effect of boundary inclination

T = 900°C
t = 528 h

No applied stress
(grip end)



Applied stress
(gauge section)



expected shift

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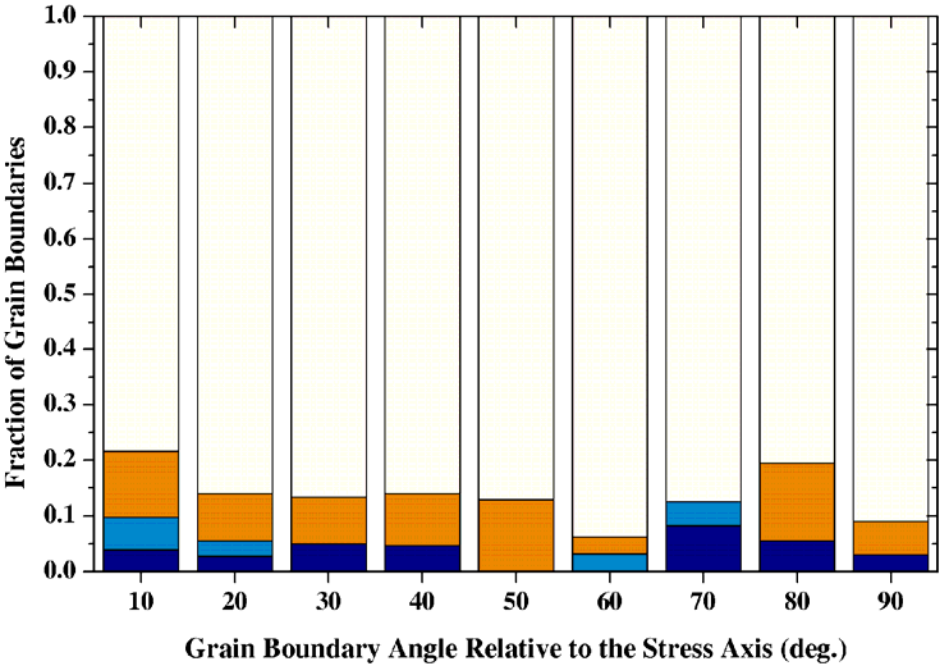
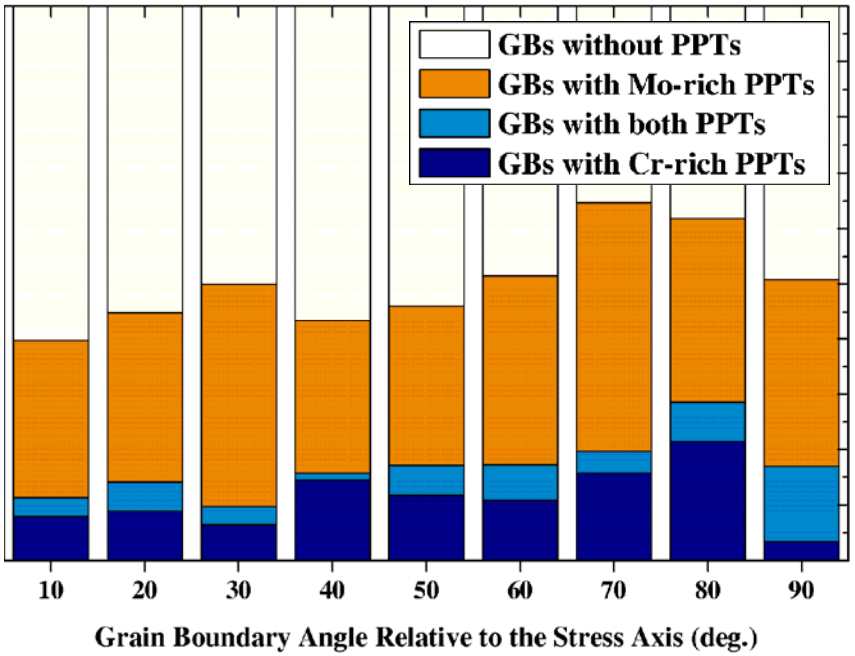
Effect of boundary inclination

T = 900°C
t = 265 h

No applied stress
(grip end)



Applied stress
(gauge section)



Why isn't the redistribution evident in this measurement?

expected shift

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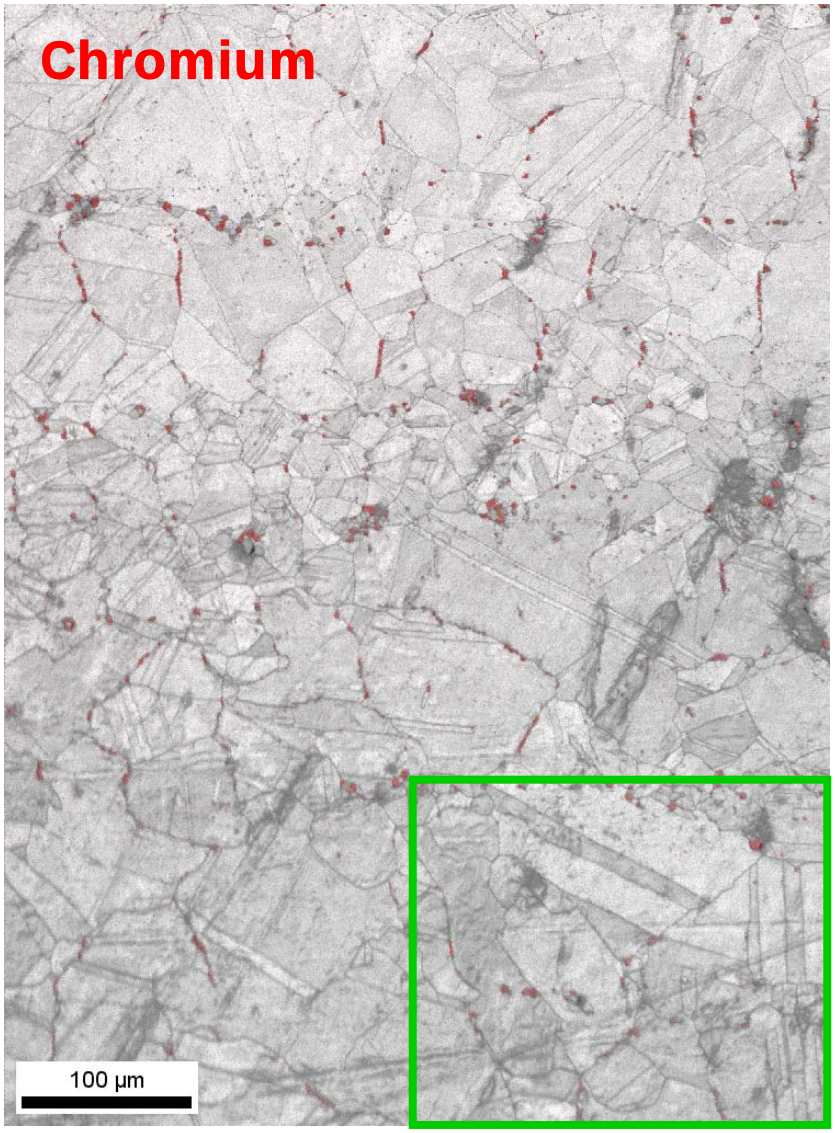
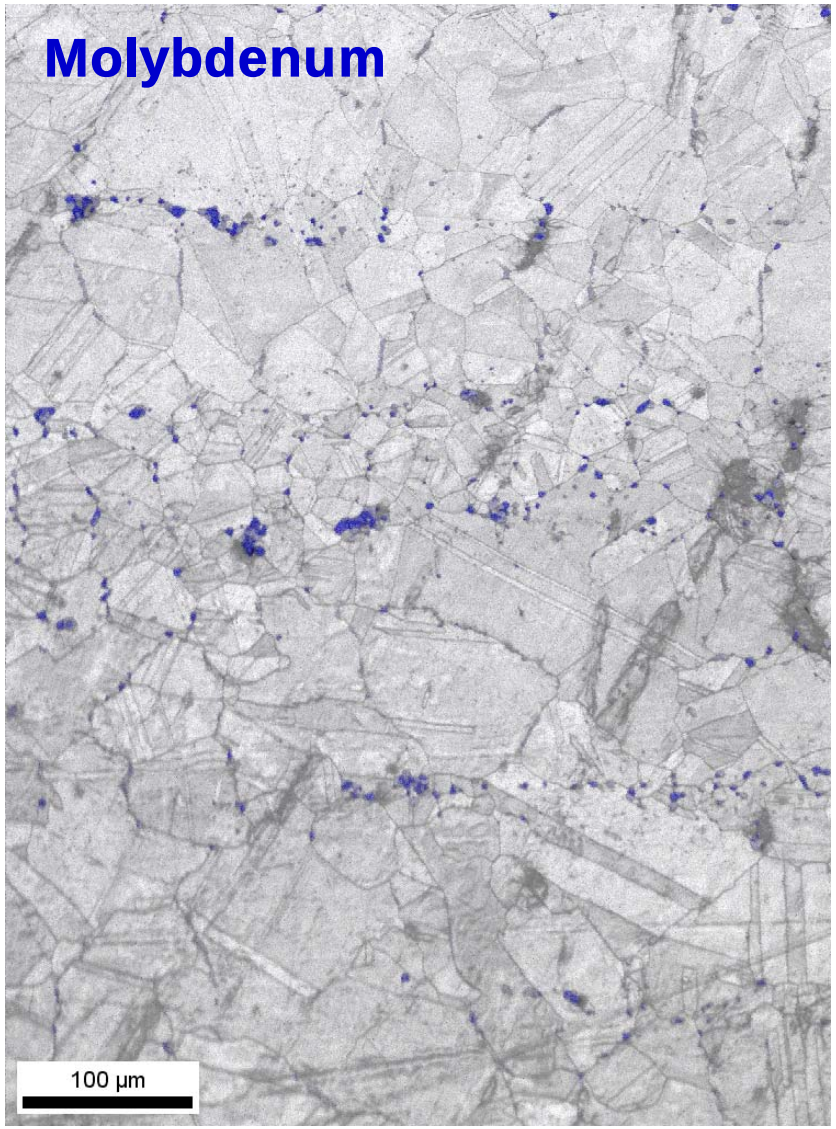
Effect of boundary inclination





Stress Axis

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Effect of boundary type (part 1)

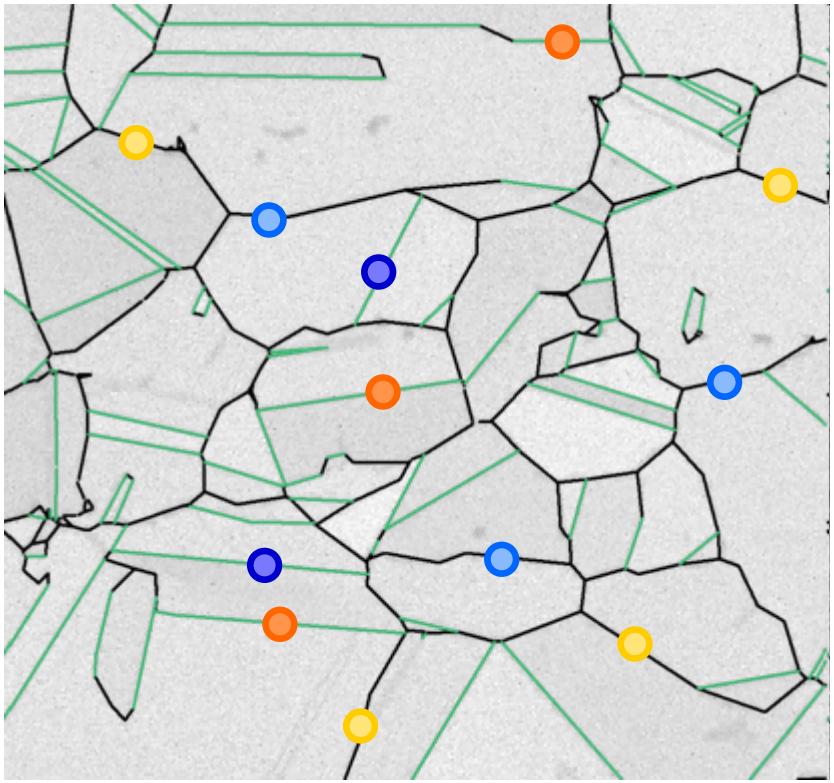
$$y(p,b) = \frac{P_{p,b}}{N_{PPT}}$$

$P_{p,b}$: # of ppts of type p on boundary type b

N_{PPT}:# of ppts in the specimen

p: ppt type (Cr-rich, Mo-rich)

b: boundary type (LA, HA, Σ3)



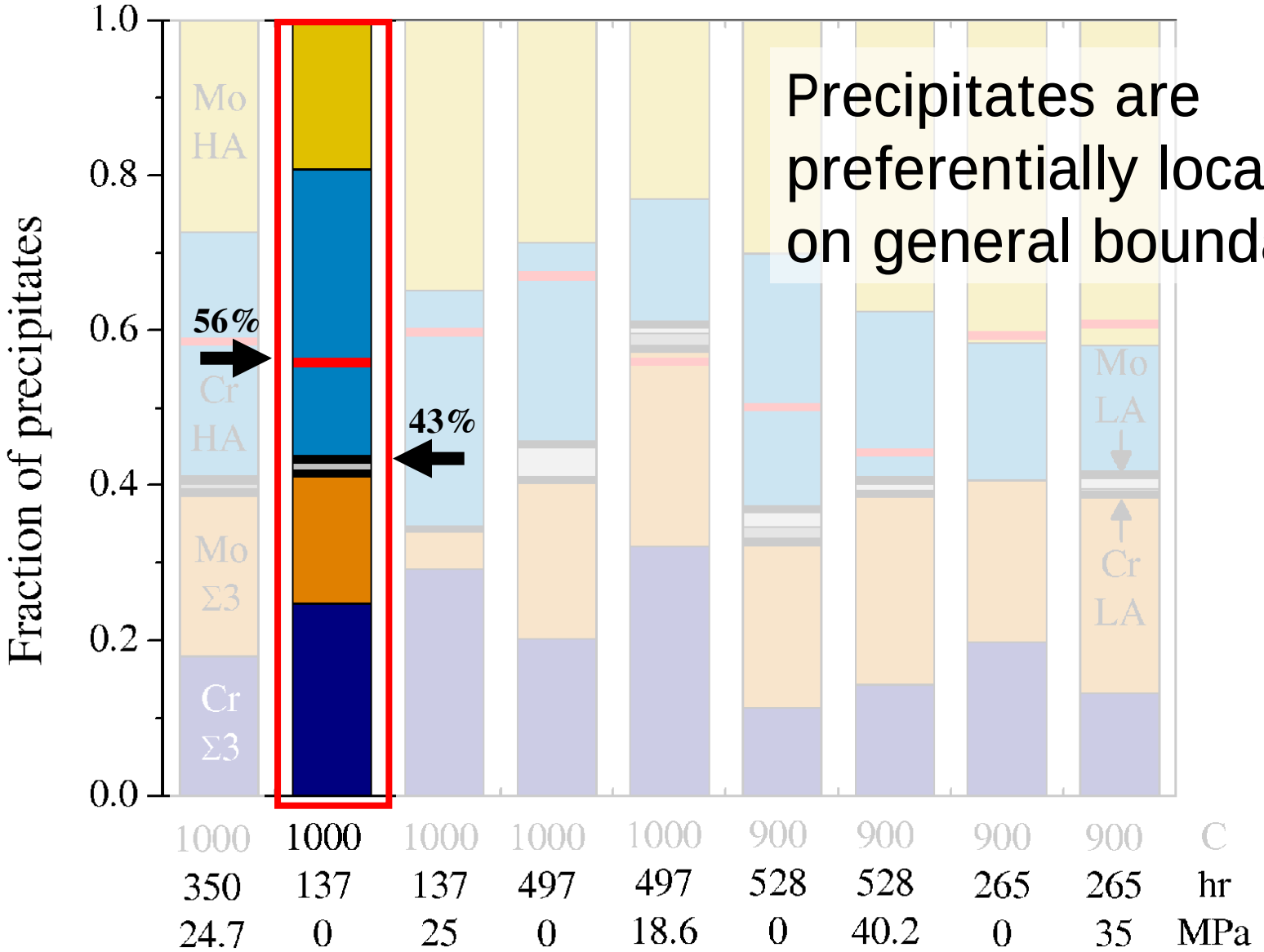
4 Mo-rich on general GBs

3 Cr-rich on general GBs

3 Mo-rich on Σ3 boundaries

2 Cr-rich on Σ3 boundaries

Effect of boundary type (part 1)



Precipitates are preferentially located on general boundaries

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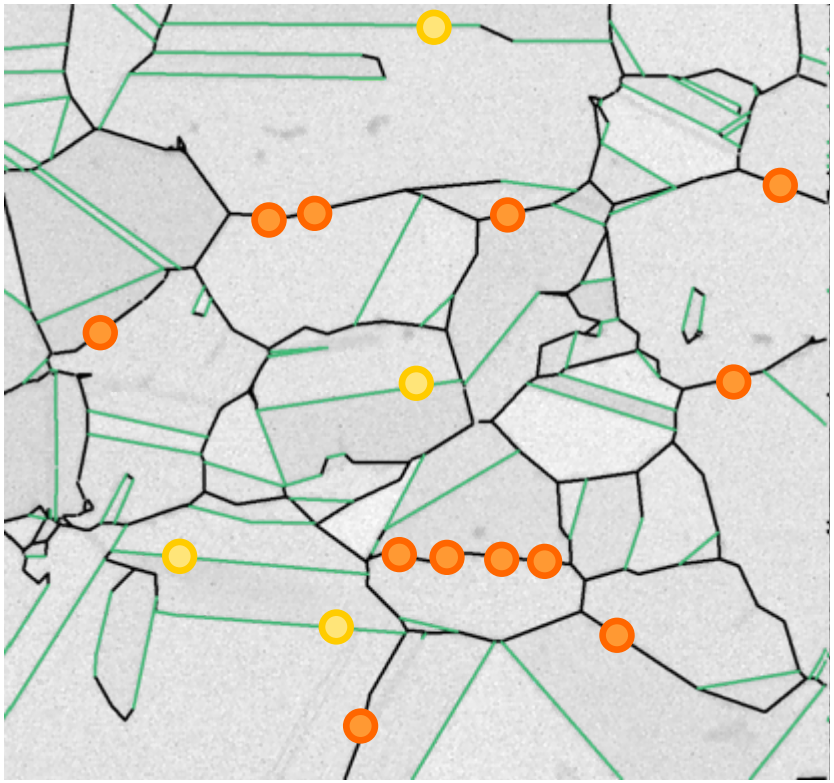
Effect of boundary type (part 2)

$$f(p,b,L) = \frac{B_{p,b,L}}{N_{GB,b}}$$



$B_{p,b,L}$: # of boundaries of type b with ppt coverage of l by ppts of type p

$N_{GB,b}$: # of boundaries of type b in the specimen

p: ppt type (Cr-rich, Mo-rich)

b: boundary type (LA, HA, Σ3)

L: length fraction of coverage

For Mo-rich ppts:

4 general GBs w/ L<0.25

3 general GBs w/ 0.25<L<0.50

1 general GBs w/ L>0.75

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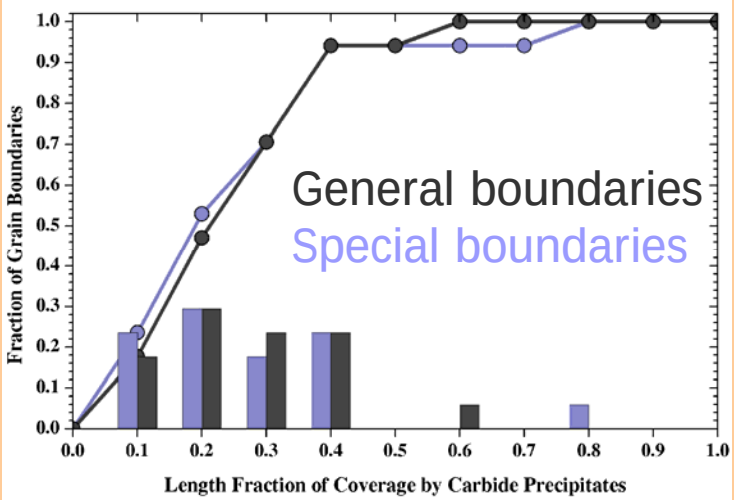
Effect of boundary type

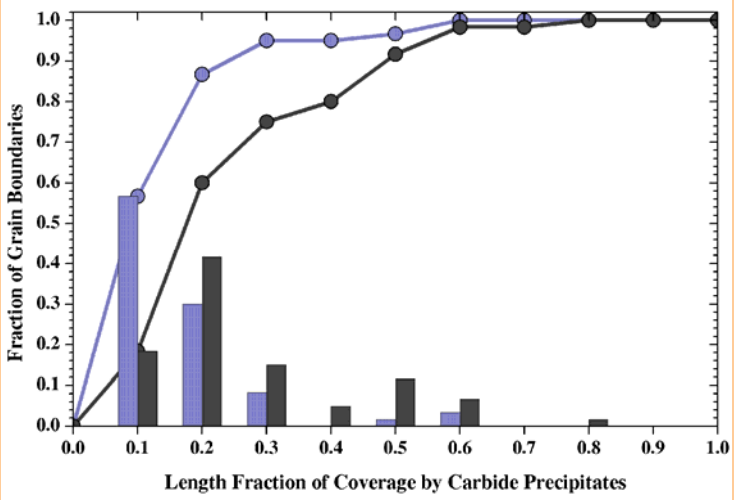
T = 1000°C
t = 497 h

Applied stress = 0

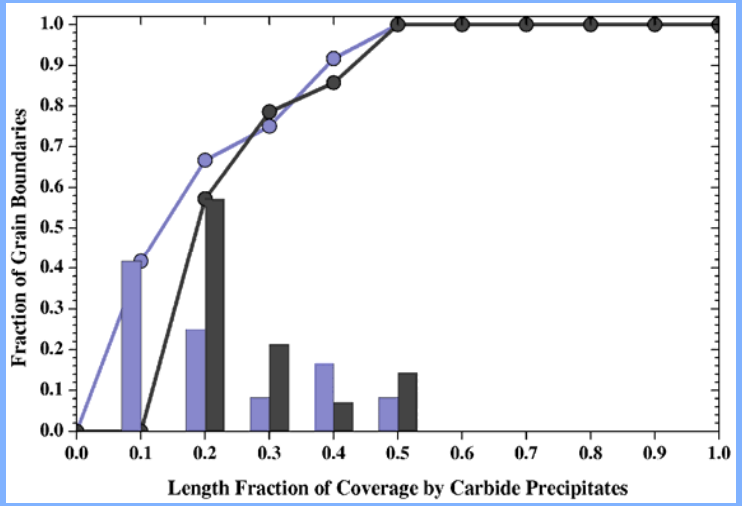
Applied stress = 18.6 MPa

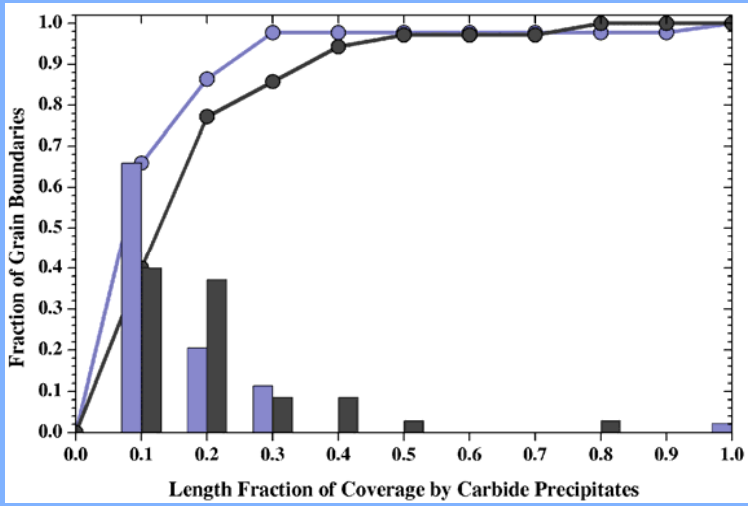
Mo-rich ppts





Cr-rich ppts





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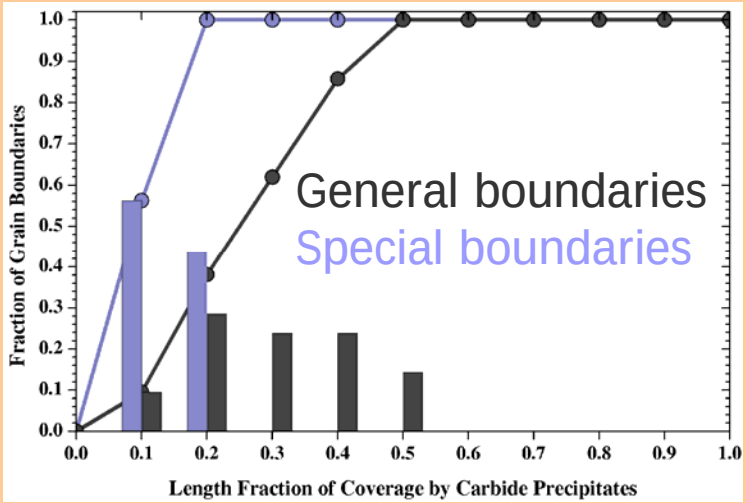
Effect of boundary type

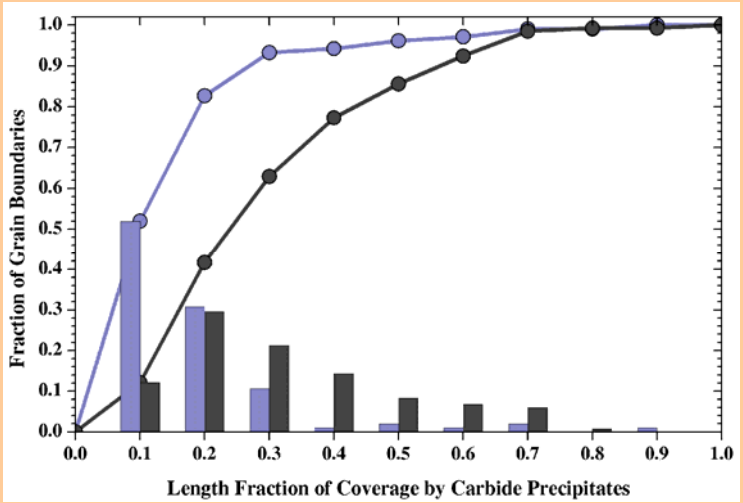
T = 900°C
t = 265 h

Applied stress = 0

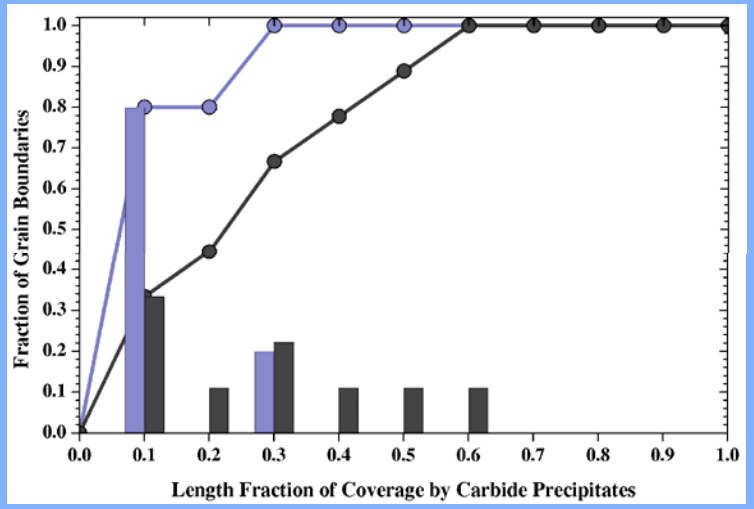
Applied stress = 35 MPa

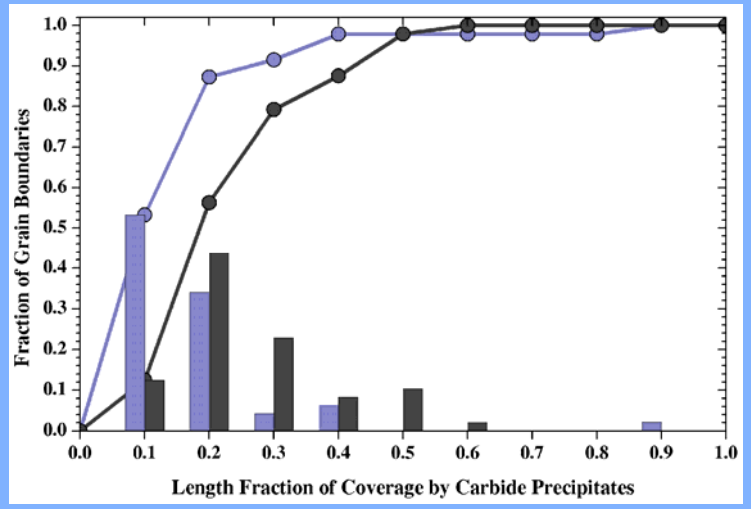
Mo-rich ppts





Cr-rich ppts





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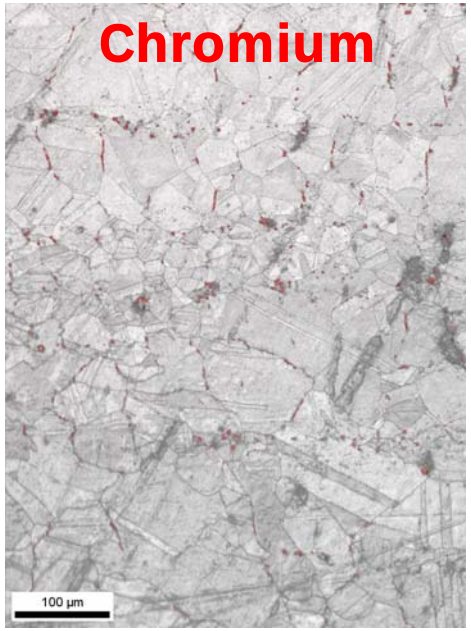
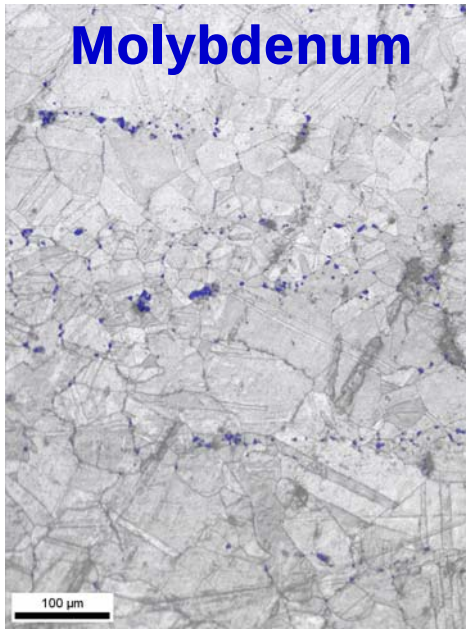
Conclusions

- After creep testing, precipitates shift to grain boundaries from grain interiors
- No noticeable effect of angle between grain boundary and stress axis
- General boundaries are more likely than $\Sigma 3$ boundaries to have more precipitates





Ongoing work

- Analyze new (larger) data sets and apply same statistical approach
- Consider the effects of stress and temperature on redistribution more closely

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