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In-Situ Tensile-Creep Testing of Structural Alloys

C.J. Boehlert

Michigan State University

Objectives

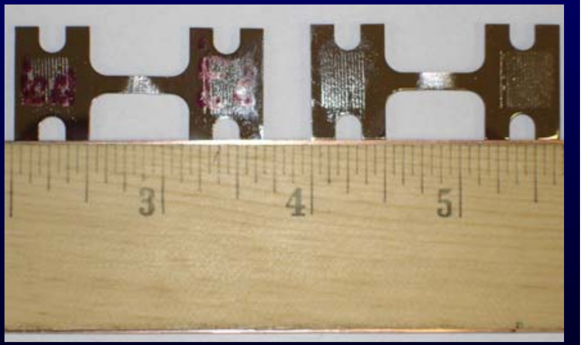
- To develop a methodolgy for performing *in-situ* elevated-temperature creep experiments on structural alloys (*in-situ* characterization includes EBSD)
- To evaluate the effect of cold rolling deformation on the microstructure and creep resistance of high-temperature structural alloys
- To understand grain boundary engineering effects on the secondary creep resistance of high-temperature structural alloys

Experimental Procedure *In-Situ* Testing

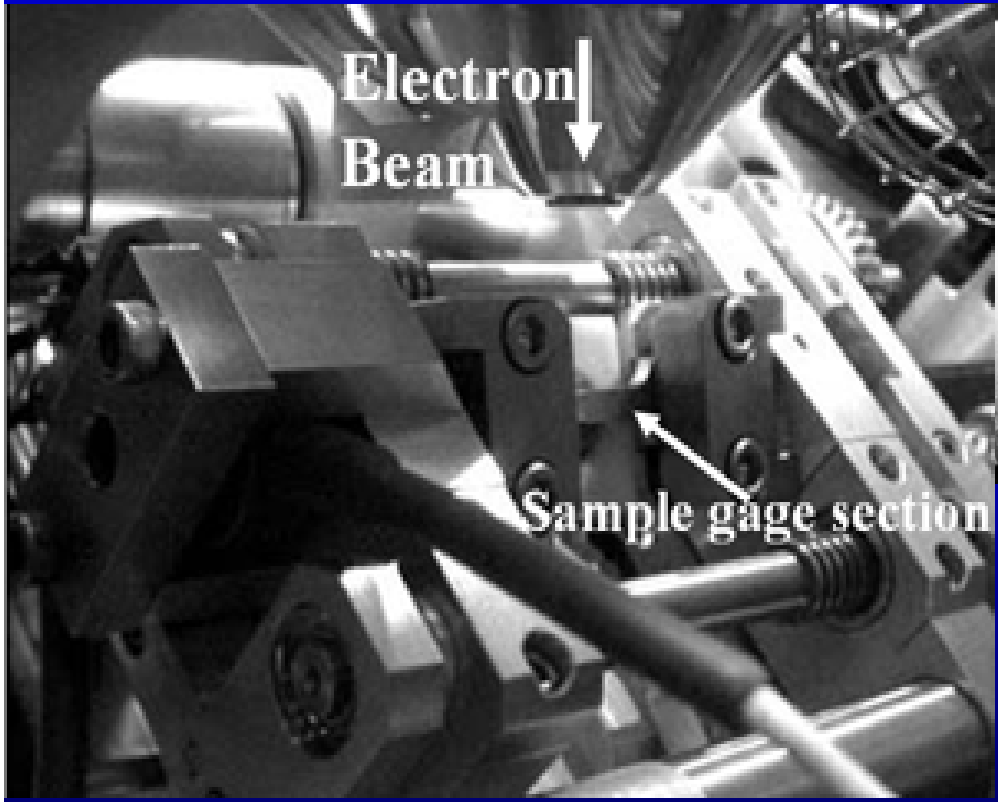
Sample ~38mm long

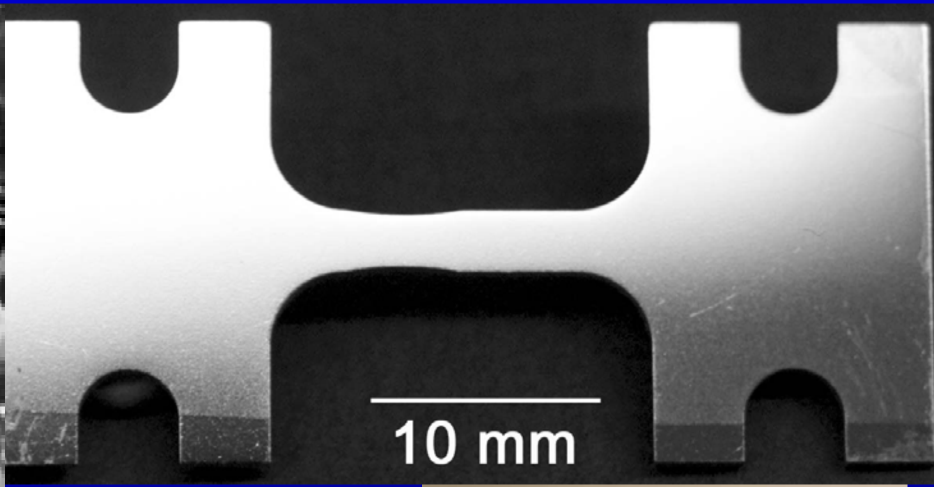
- **Specimens EDM machined to custom geometry**
- **Screw driven actuator: Load or displacement controlled**
- **Load capability = 1000lbs; smaller load cells available**
- **Tensile Stage can tilt 45 degrees**
- ***In-situ* EBSD possible through SEM stage tilt,**
  Hikari Camera used: 150pts/sec indexing rate.
  It took 20 minutes to map a 200umx200um area (1kX magnification)
- **Sample heated using 6mm diameter W-based radiant heater/power supply**
- **Steel grips are not heated**
- **Vacuum maintained at 10^{-6} torr throughout experiment (longest~14days)**
- **Heater cooled through Cu lines**

  Open Bath Chiller slowly pumping distilled water at 20C
- ***In-situ* SE imaging throughout experiment (tilt-corrected to remove foreshortening)**
- **Local Strain Mapping may be accomplished through image analysis**
- **Type K thermocouples fed through SEM port cover**
- **Admet Software recording+displaying time, grip displacement, load continuously**
- **Gage section strain has been measured with strain gage attached to specimen face (not imaged)**



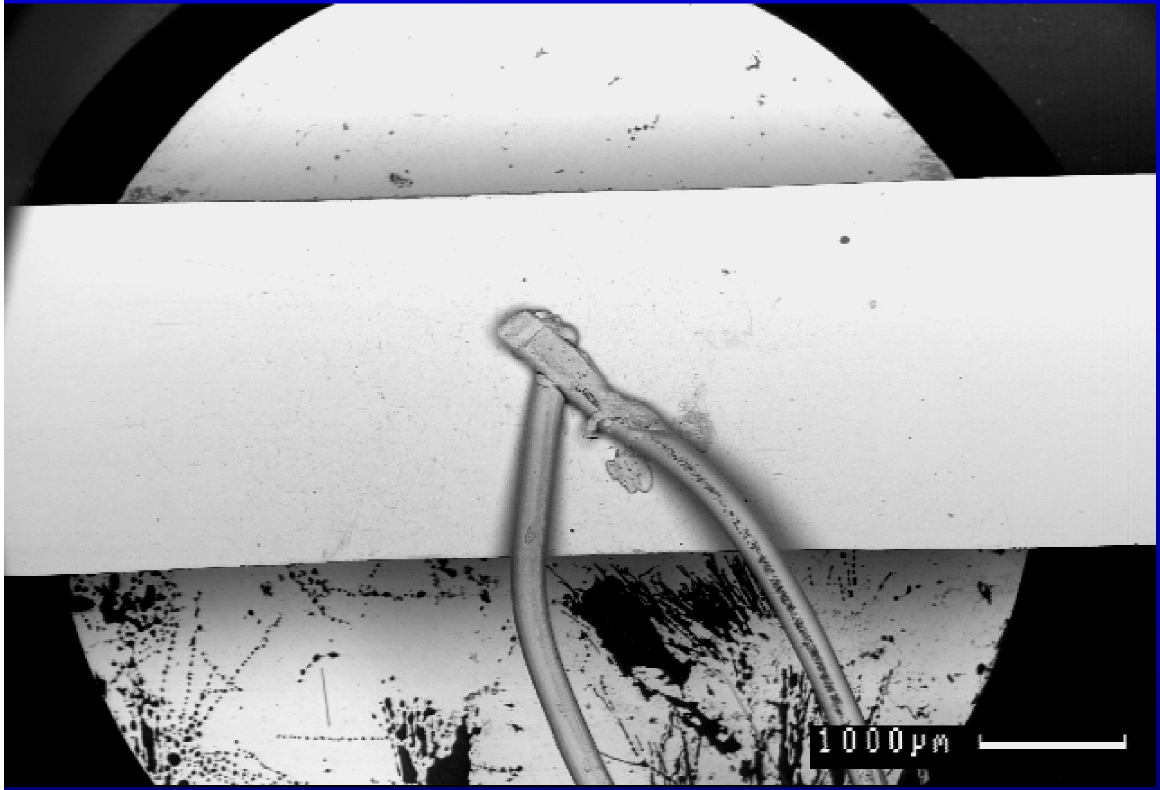
Experimental Procedure *In-Situ* Testing

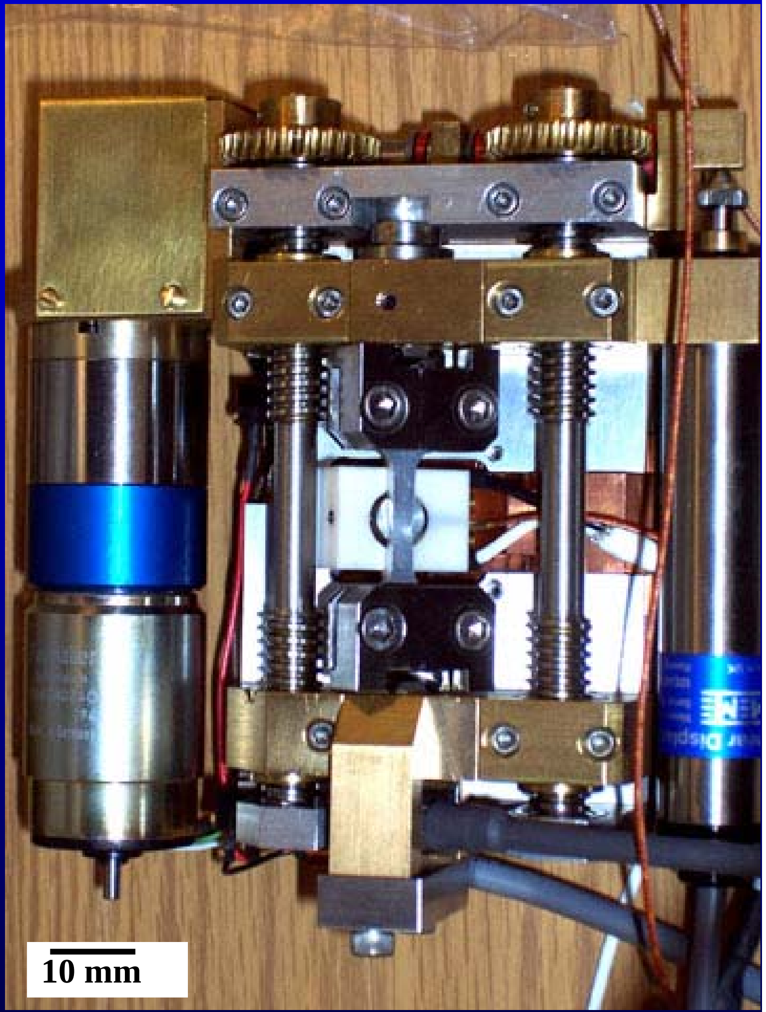




A specially-designed specimen was used along with an Ernest Fullam Tensile Stage to perform creep experiments at temperatures up to 760°C and loads up to 1000lbs. EBSD orientation maps can be performed both before and after creep deformation as well as *during* the creep deformation experiment.

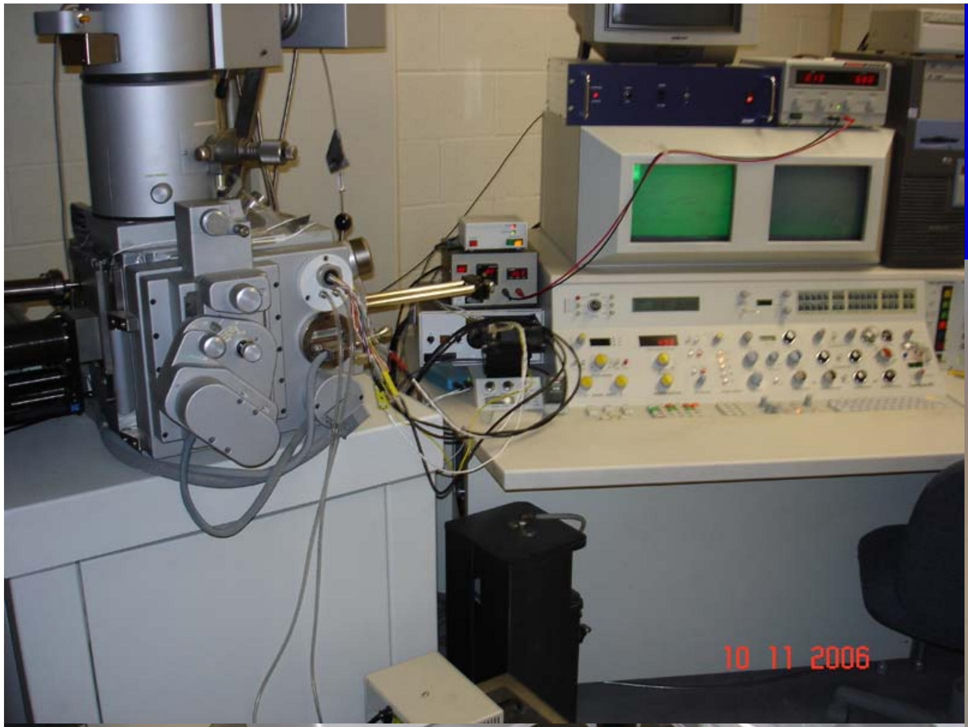
Temperature Measurement

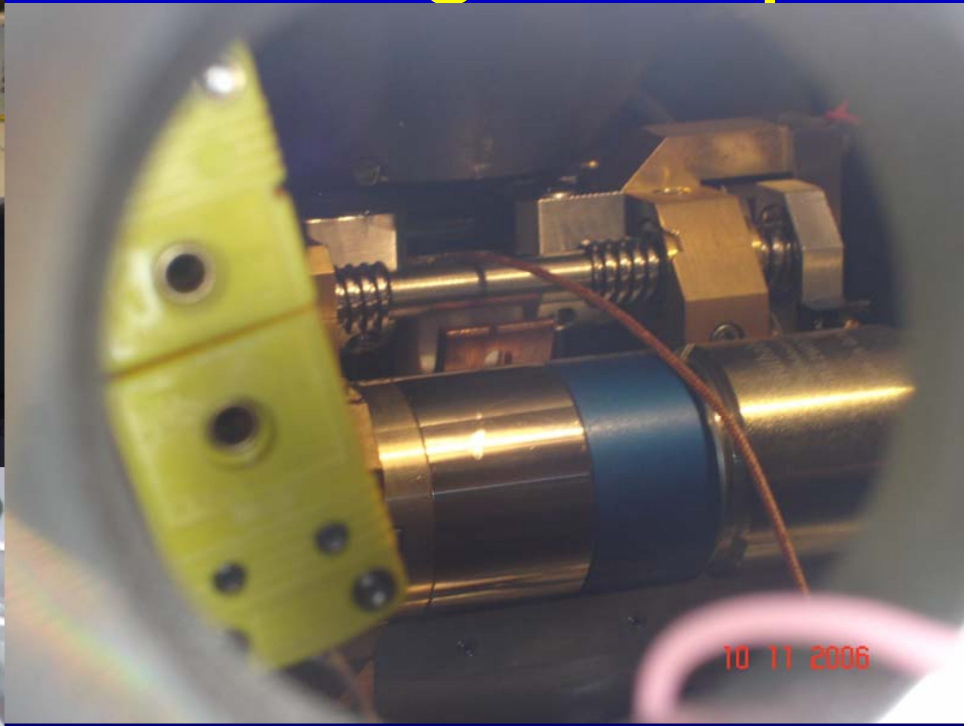


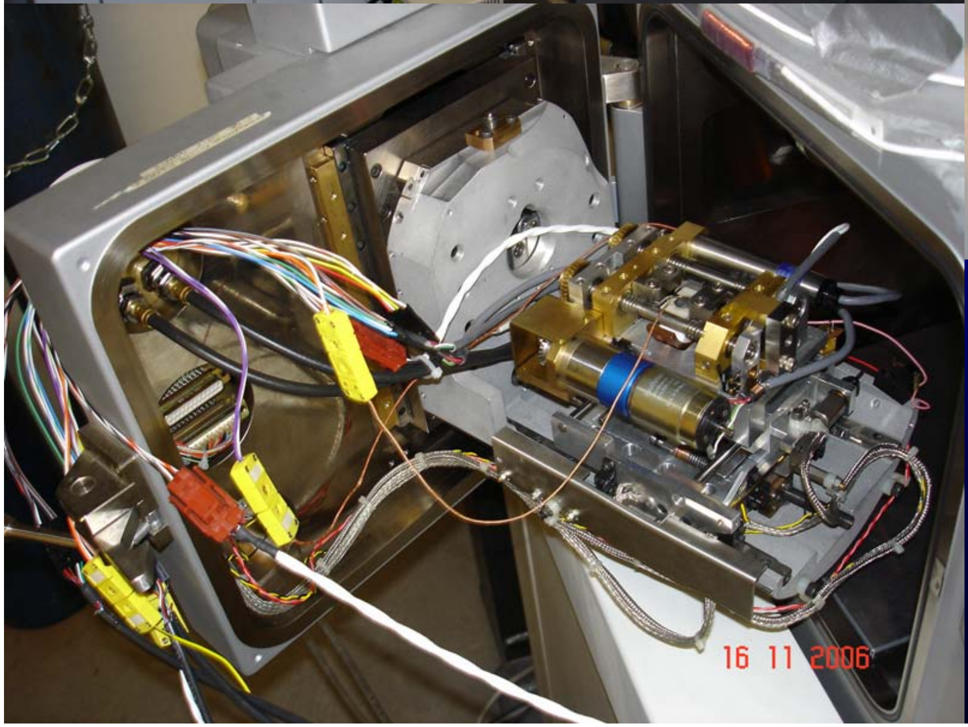


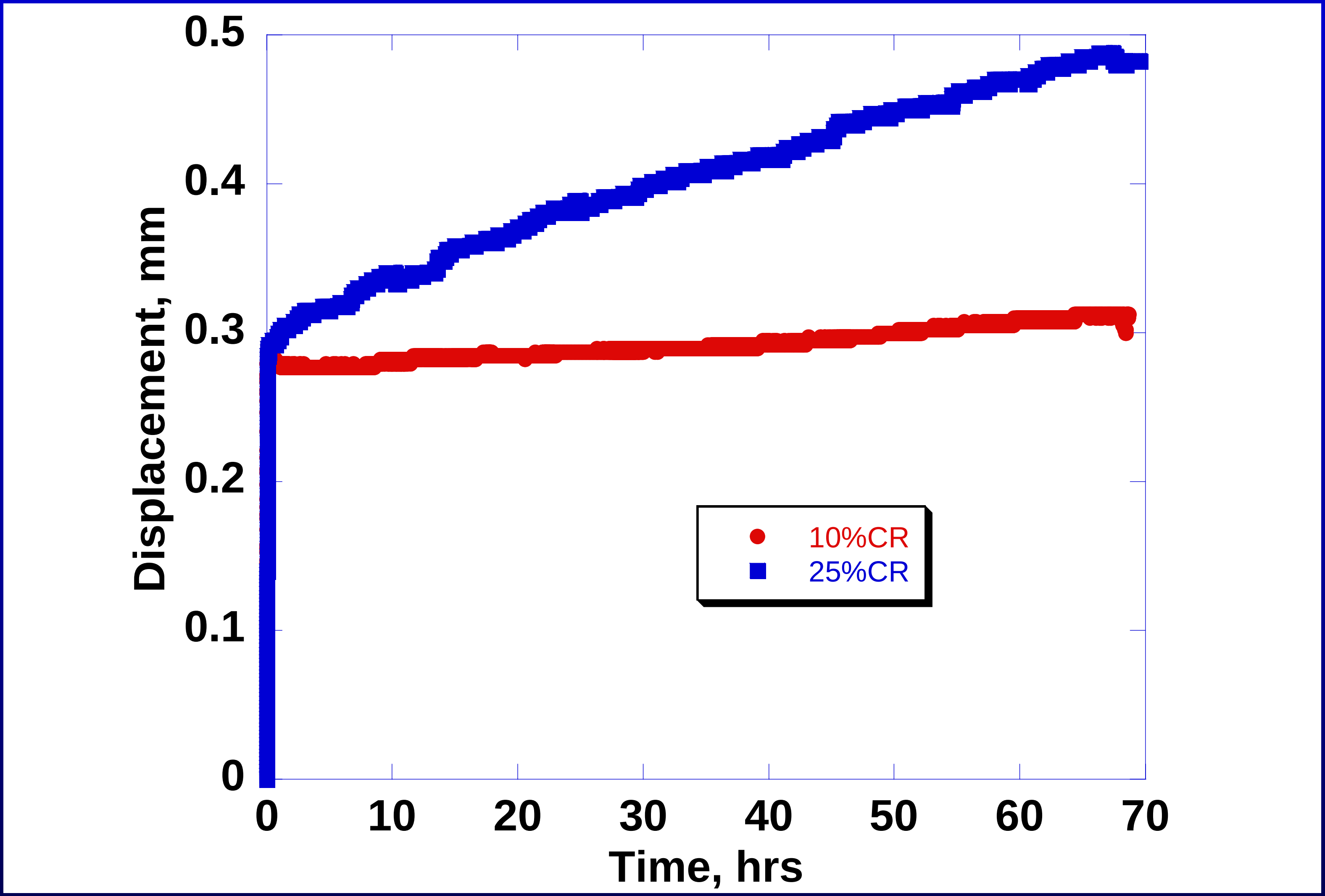
6mm diameter W-based heater was located just below the gage section of the sample. Fine-gage type-K TC monitored the temperature and SEM BSE micrographs were taken within the heated section during creep.

In-situ Creep Testing Setup



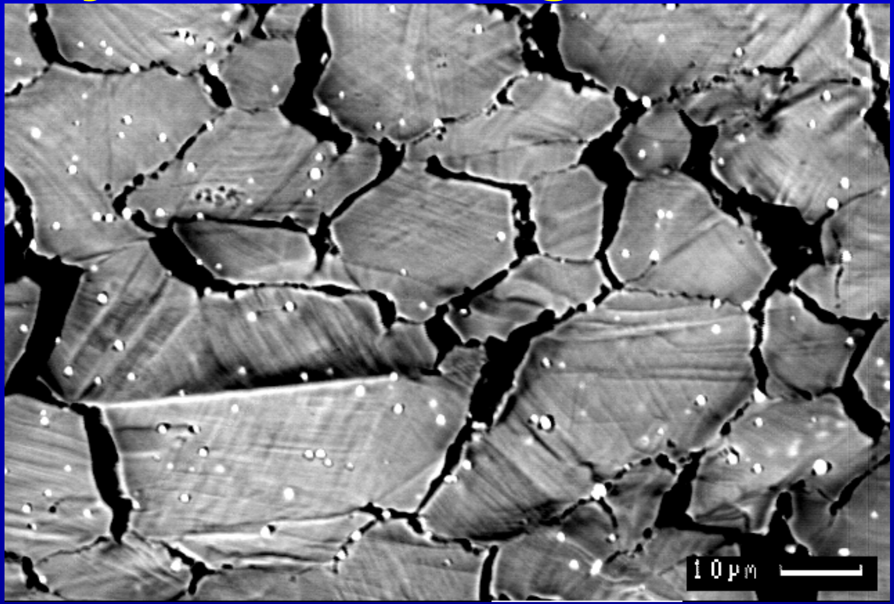


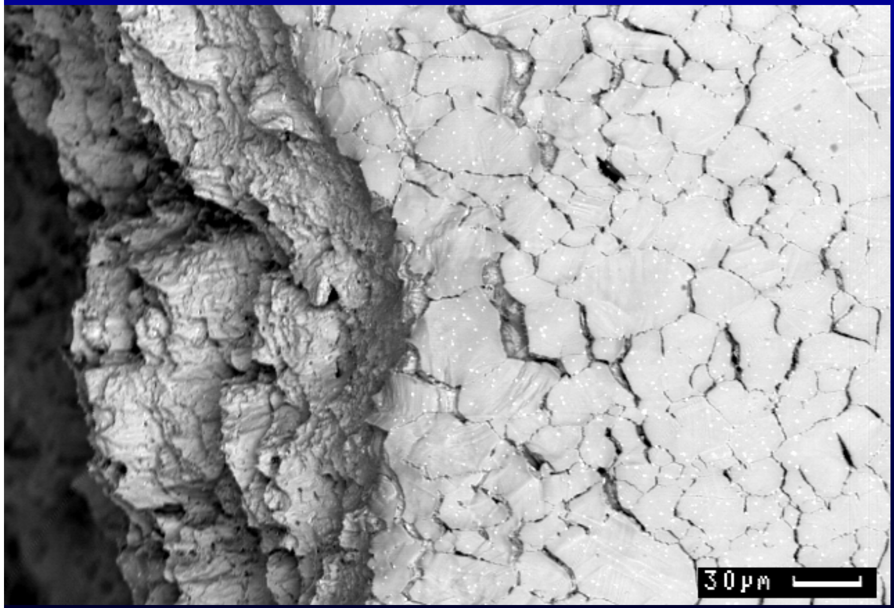


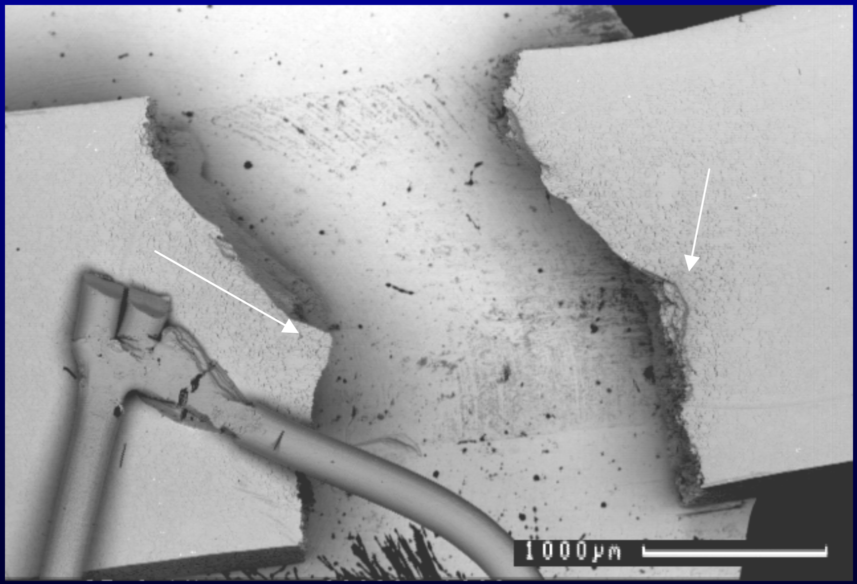


Displacement versus time curves for creep experiments performed at T=650°C and σ=300MPa.

Grain Boundary Cracking

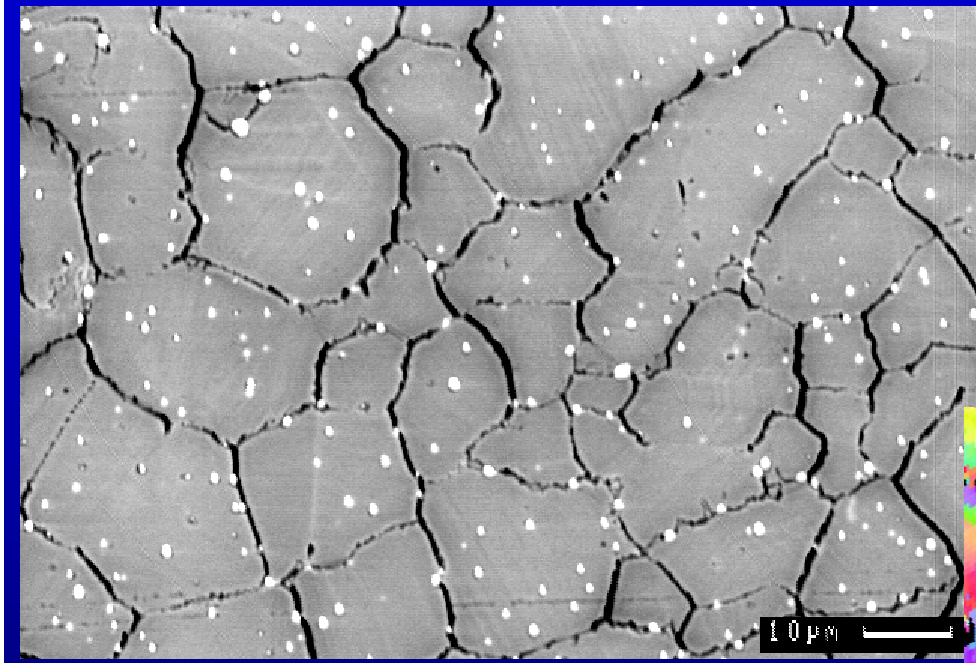


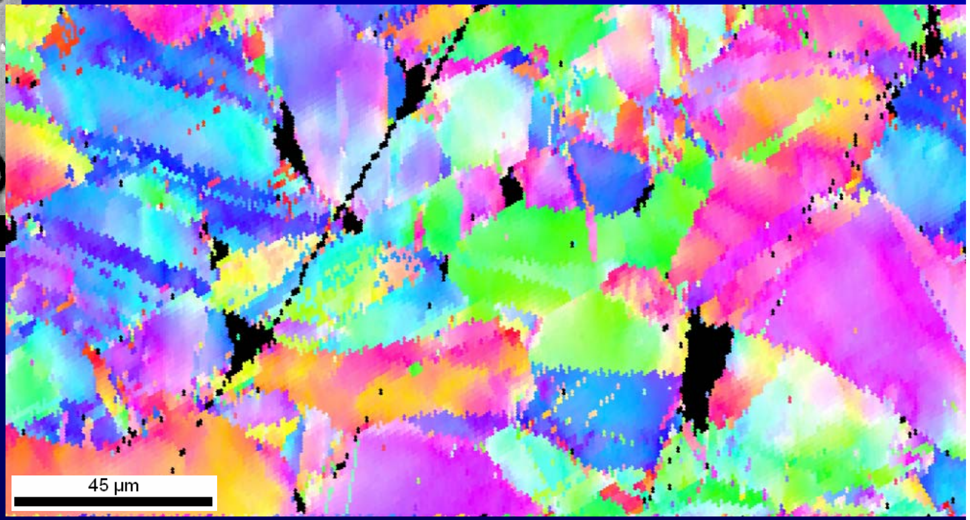




35% CR UD 188

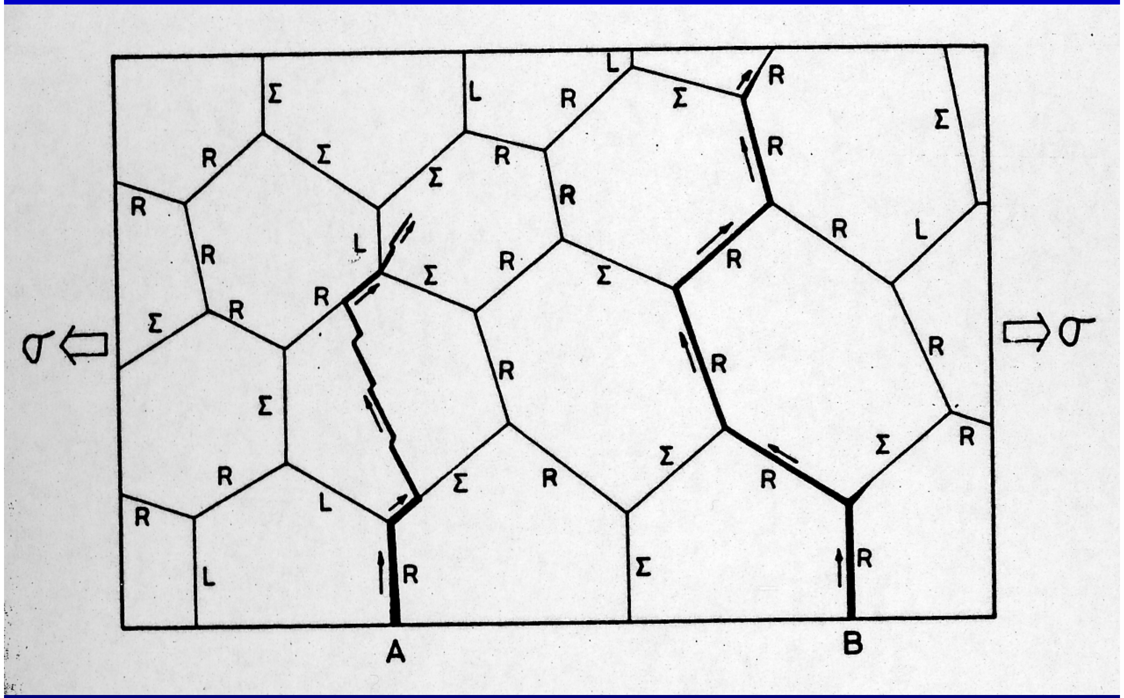
Cracking at HABs

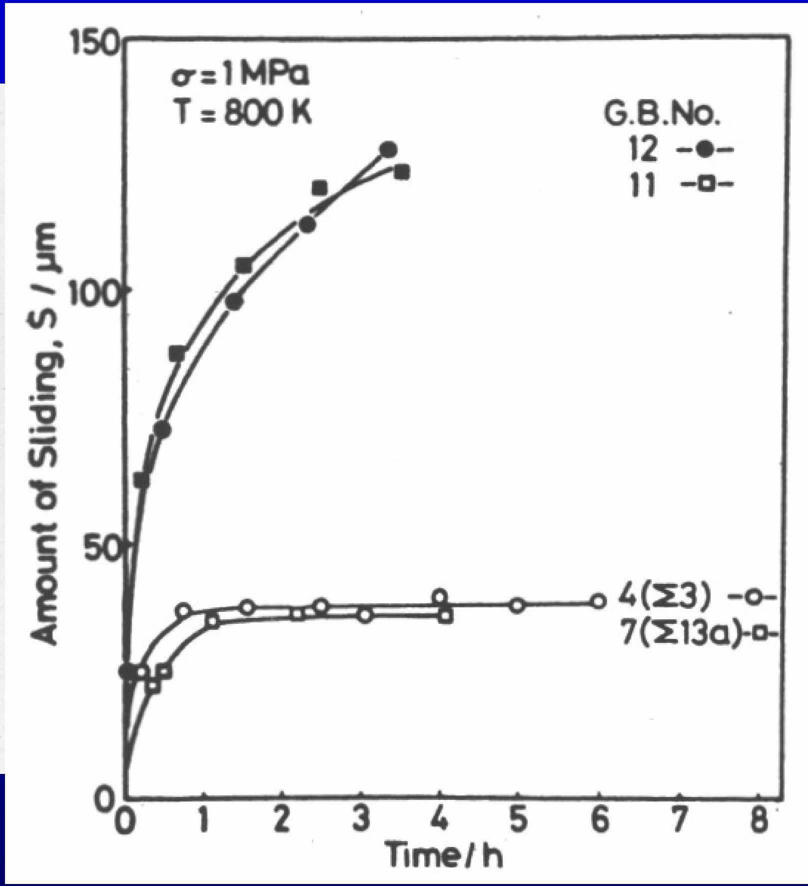




BSE SEM image of a 25% cold rolled sample that was tested *in-situ* at 650°C and 300MPa and an EBSD map of a 25% cold rolled sample that had undergone a creep rupture experiment at 815°C and 165MPa and exhibited over 20% strain to failure. This area was from a subsurface polished section near the fracture surface. Substantial lattice curvature is evident in the color gradients within most grains. The loading direction was horizontal.

Effect of Grain-Boundary Structure on the Fracture Process



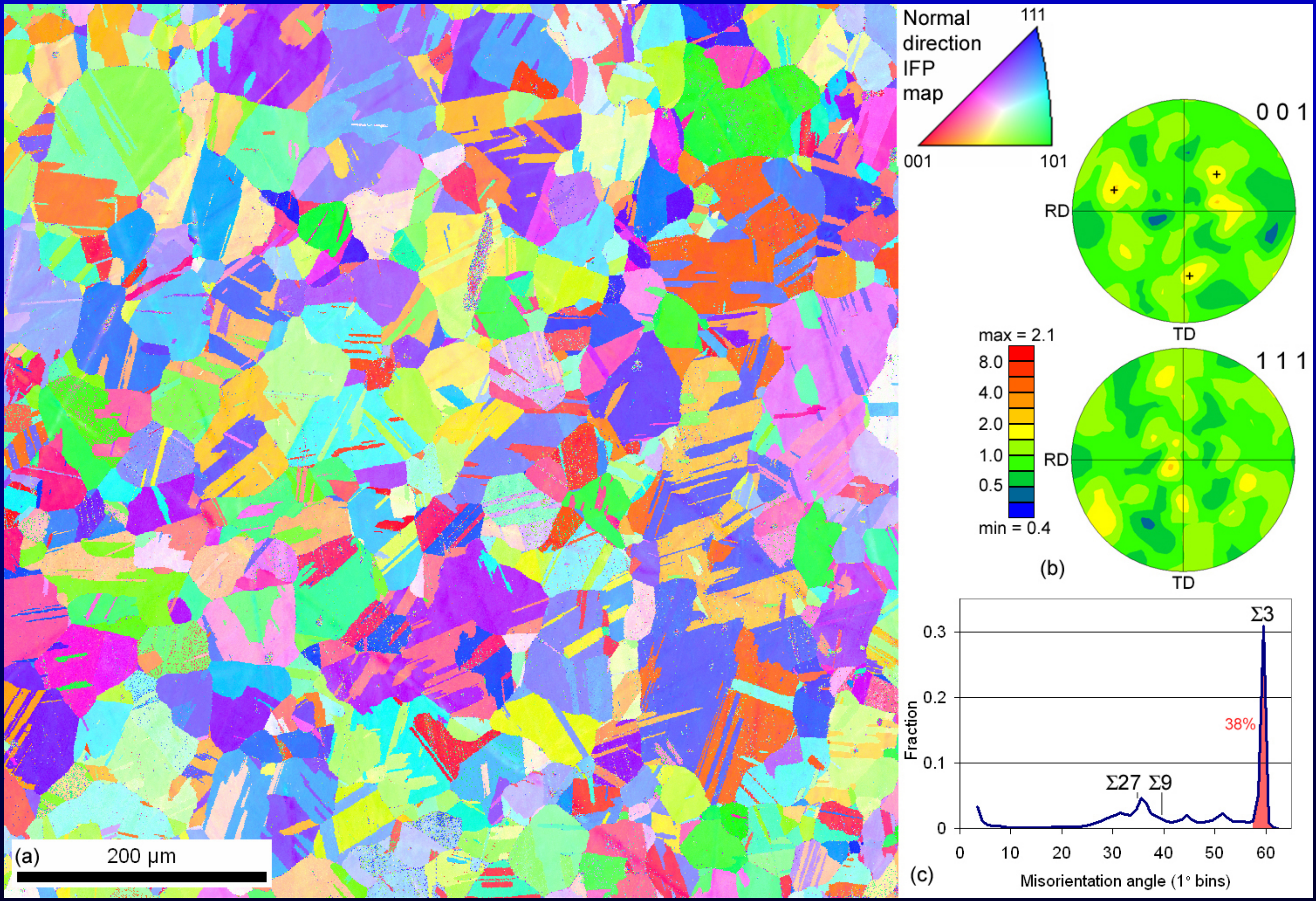


Random boundaries are susceptible to intergranular degradation. Increased proportion of special boundaries can improve fracture resistance by preventing intergranular degradation and decreasing diffusion across grain boundaries

T. Watanabe, 'An Approach to Grain Boundary design for strong and ductile for strong and ductile polycrystals', Res Mechanics, 11(1984) pp. 47-84.

Grain Boundary Character Distribution

- **Weak texture, but lots of annealing twins occur in Udimet alloy 188 after TMP**

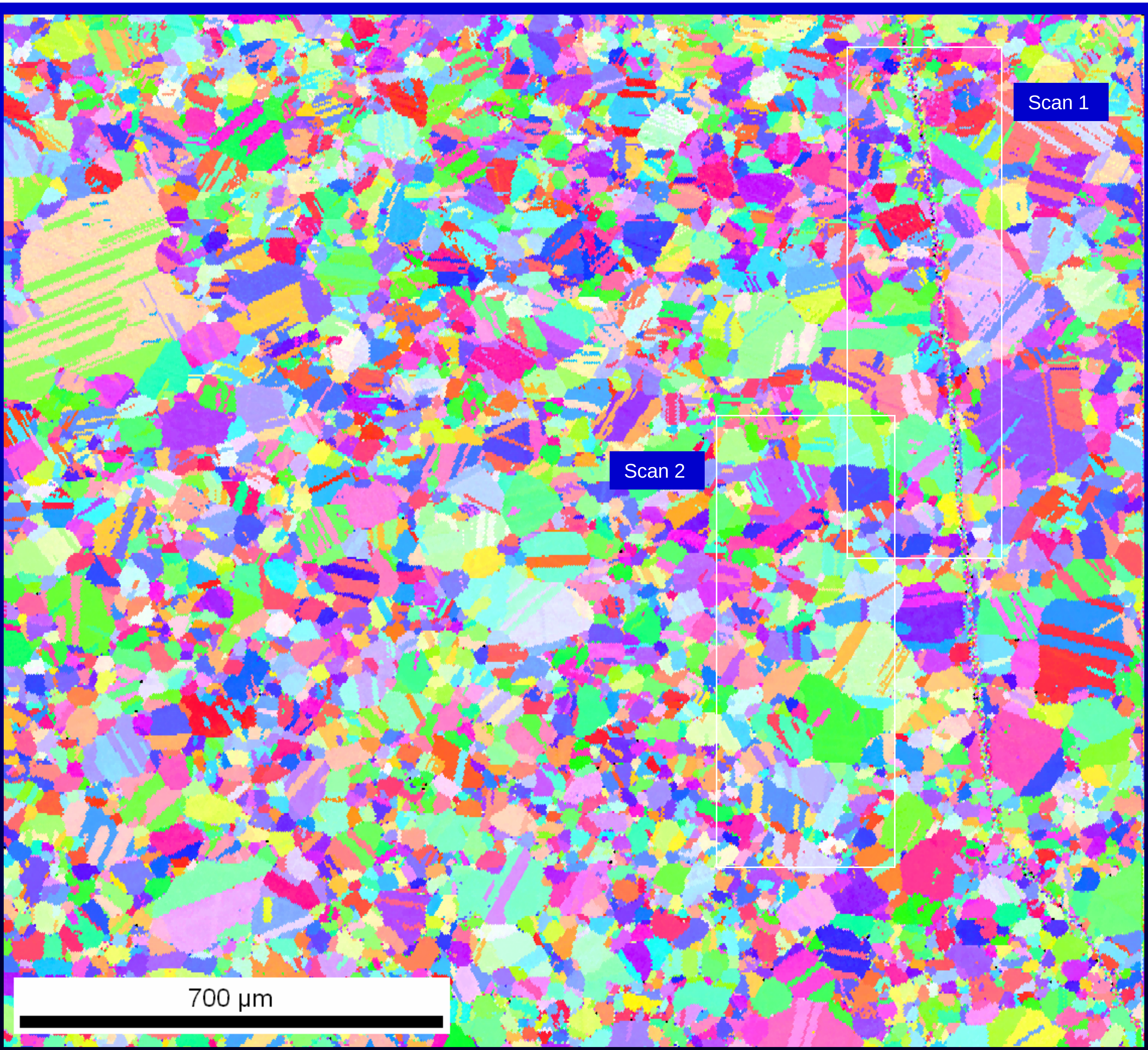


Ex-Situ EBSD Analysis

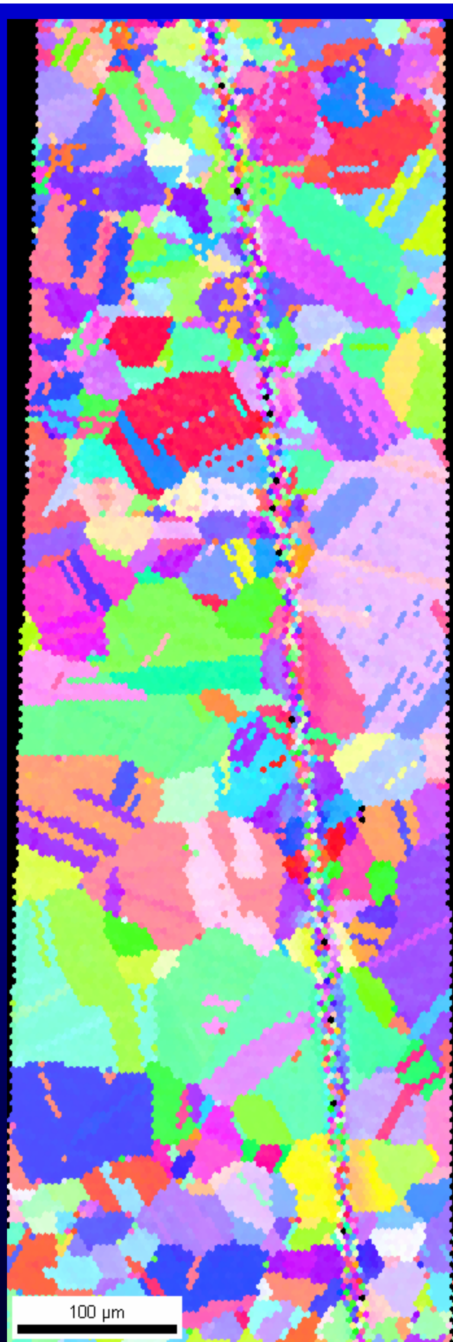
- EBSD Maps were performed on the specimen prior to testing
- Fiducial markings were positioned so that after creep testing the same areas could be mapped after creep testing to a given strain
- The sample was tested so that the sample surface was perpendicular to the incident beam.
- After the experiment the sample was removed from the tensile stage and placed on a separate holder then EBSD mapping was performed.
- Additional Creep Experiments could be performed on the same sample, however the sample would need to undergo heating and loading each time.

Before – Scan 1

4um step size



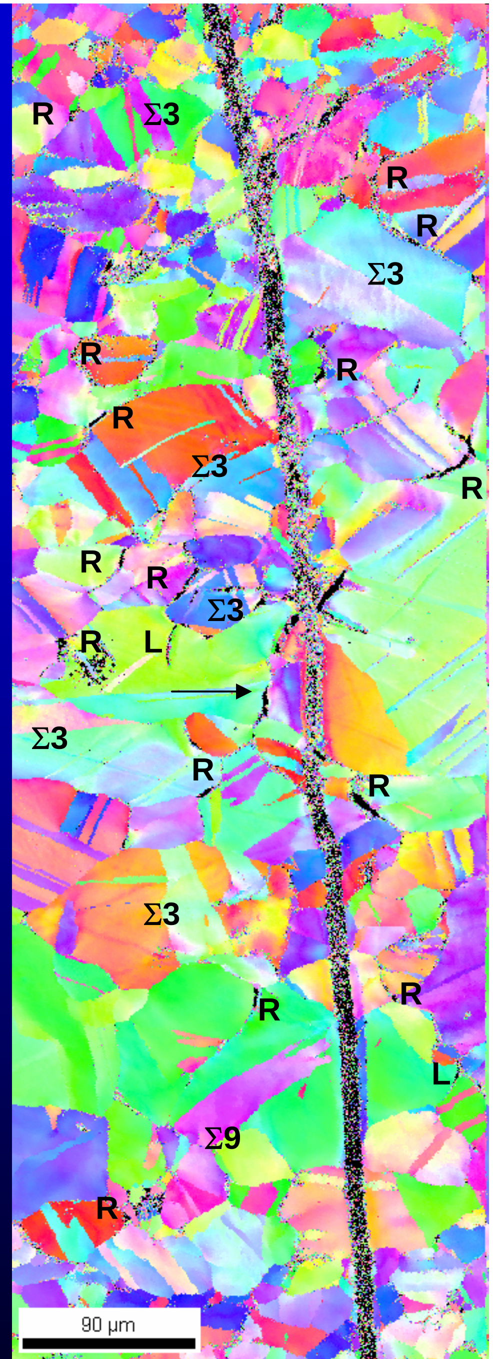
Before – Scan 1;
4um step size



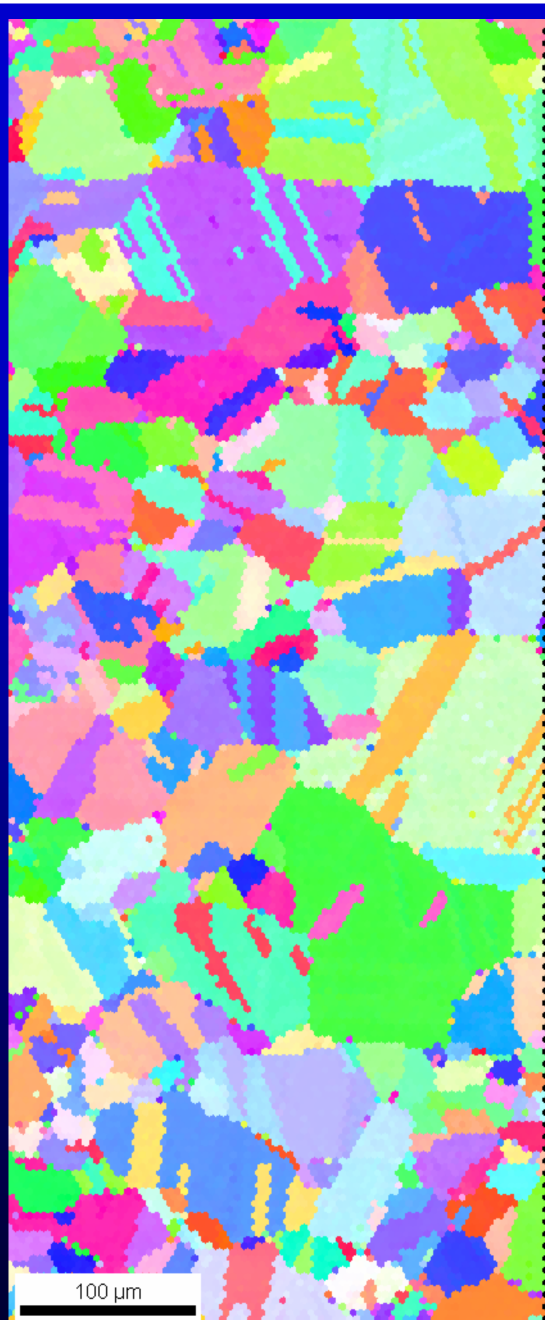
After – Scan 1;
0.75um step size

Of the more than 20 cracked boundaries only one was a special boundary (Σ9 see black arrow). All others were GHABs (R). Two were LABs.

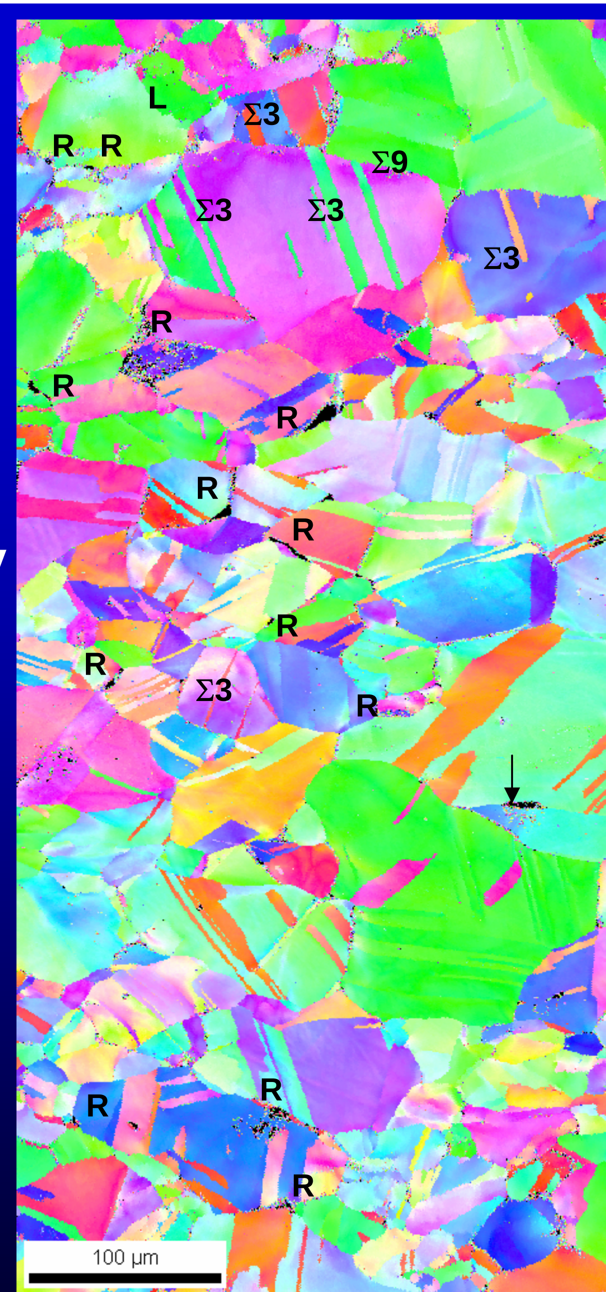
06-17 35%cr
760°C,220MPa;
4-10%cr str



Before – Scan 2; 4um step size



After – Scan 2; 0.75um step size



Of the more than 20 cracked boundaries only one was a special boundary (Σ3 see black arrow). All others were GHABs (R).

06-17 35%cr
760°C,220MPa;
4-10%cr str

R | L | Σ3

TD

RD

Gray Scale Map Type: Image Quality
34.175...210.917 (34.175...210.917)

Color Coded Map Type: <none>

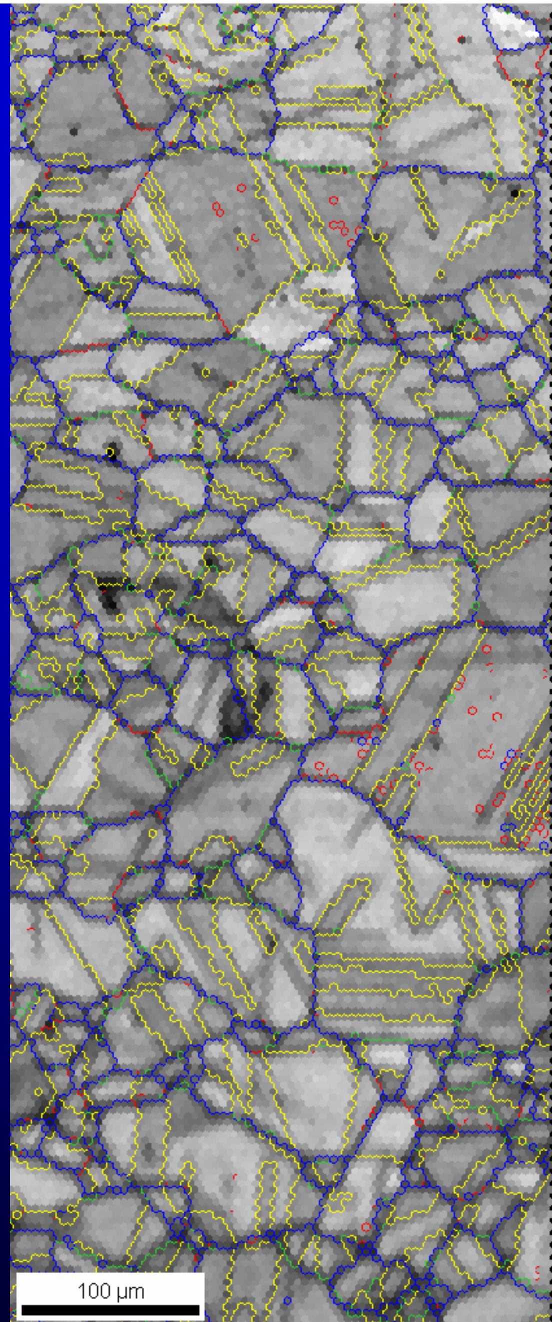
Boundaries: Rotation Angle

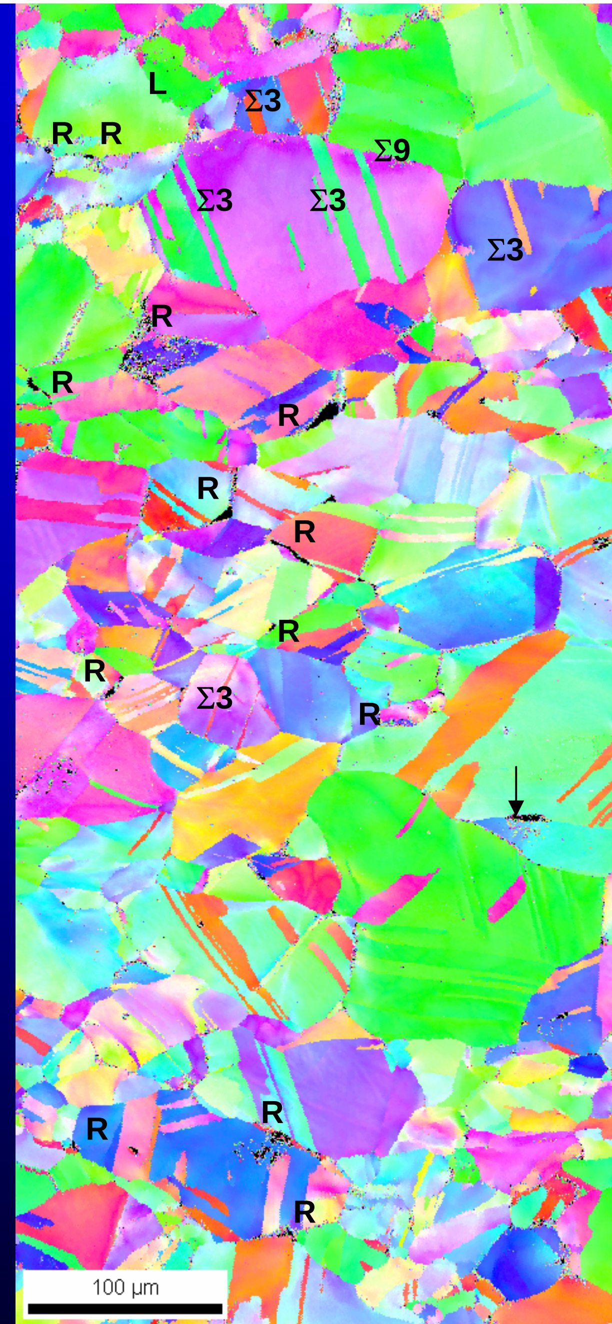
	Min	Max	Fraction	Number	Length
blue	15°	180°	0.949	11824	2.73 cm
red	2°	15°	0.051	641	1.48 mm

Boundaries: CSL

	Sigma	Tolerance	Fraction	Volume	MDF Value	Number	Length
yellow	3	8.66	0.489	0.0176	27.86	6101	1.40896 cm
green	5	6.71	0.002	0.0123	0.18	28	64.6632 microns
green	7	5.67	0.004	0.0099	0.42	52	120.089 microns
green	9	5.00	0.030	0.0102	2.92	370	854.478 microns
green	11	4.52	0.005	0.0075	0.69	65	150.111 microns
green	13a	4.16	0.002	0.0029	0.69	25	57.735 microns
green	13b	4.16	0.003	0.0039	0.82	40	92.376 microns
green	15	3.87	0.004	0.0094	0.41	48	110.851 microns
green	17a	3.64	0.000	0.0020	0.08	2	4.6188 microns
green	17b	3.64	0.004	0.0039	0.96	47	108.542 microns
green	19a	3.44	0.004	0.0033	1.33	55	127.017 microns
green	19b	3.44	0.003	0.0022	1.38	38	87.7572 microns
green	21a	3.27	0.000	0.0019	0.17	4	9.2376 microns
green	21b	3.27	0.001	0.0057	0.14	10	23.094 microns
green	23	3.13	0.001	0.0050	0.13	8	18.4752 microns
green	25a	3.00	0.000	0.0011	0.22	3	6.9282 microns
green	25b	3.00	0.002	0.0044	0.46	25	57.735 microns
green	27a	2.89	0.005	0.0020	2.59	63	145.492 microns
green	27b	2.89	0.005	0.0039	1.21	59	136.255 microns
green	29a	2.79	0.010	0.0009	10.88	119	274.819 microns
green	29b	2.79	0.001	0.0035	0.23	10	23.094 microns
	summary	-	0.575	0.1133	5.08		

*For statistics - any point pair with misorientation exceeding 2° is considered a boundary (total number = 12465, total length = 2.88 cm)





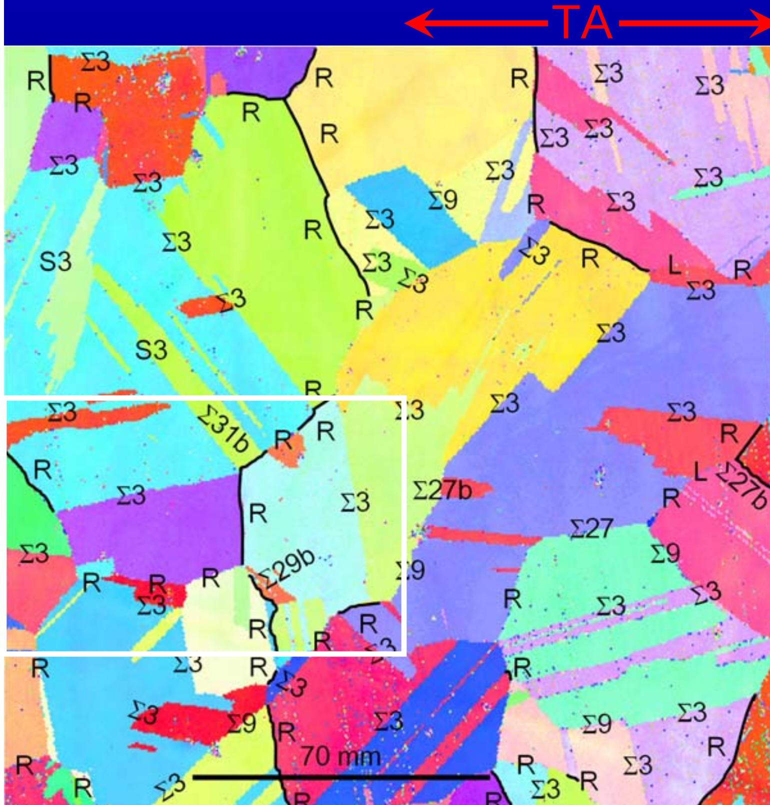
Before – Scan 2; 4um step size

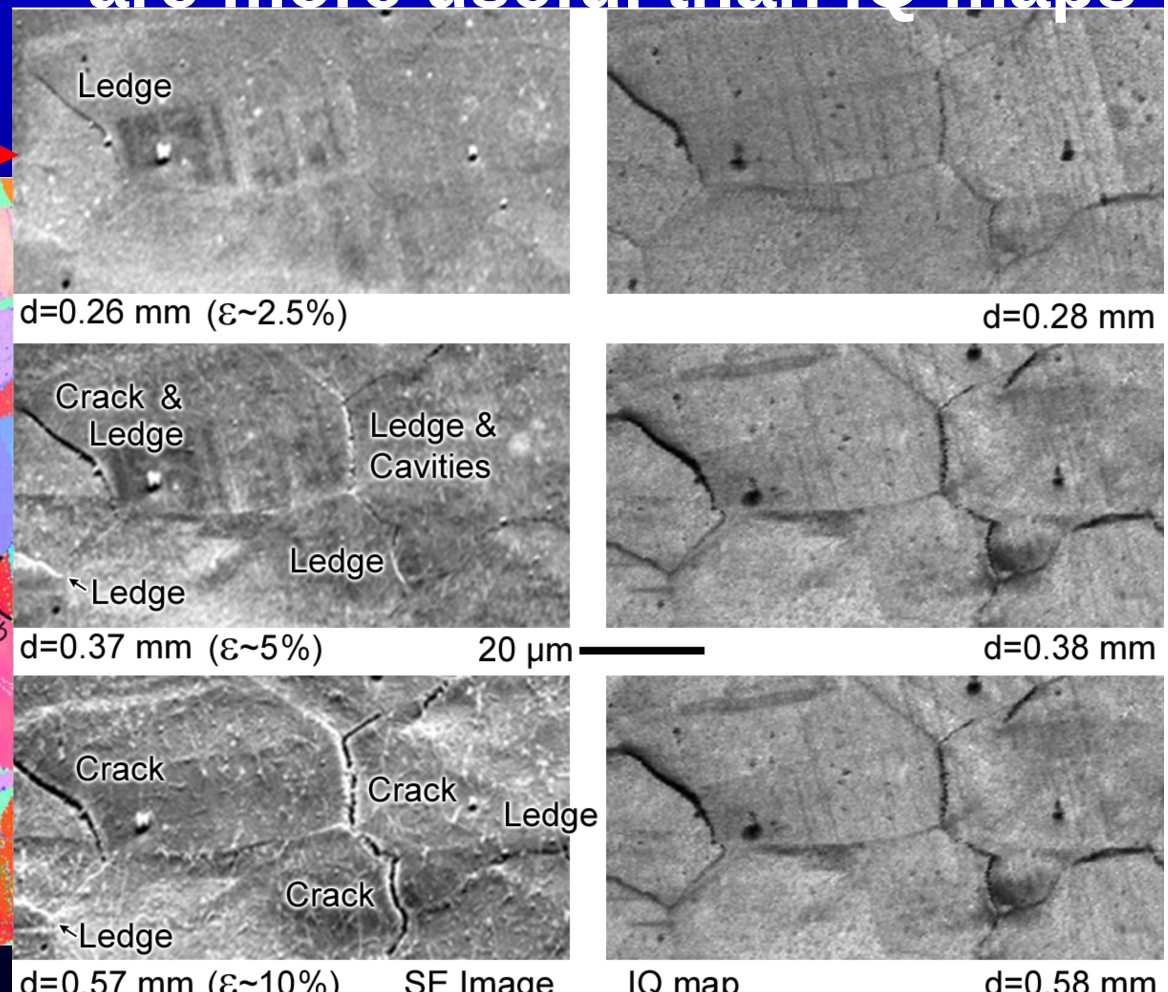
35%cr UD 188
760°C, 220MPa;
4-10%cr str

Almost no cracks on Σ boundaries, about half of random boundaries cracked

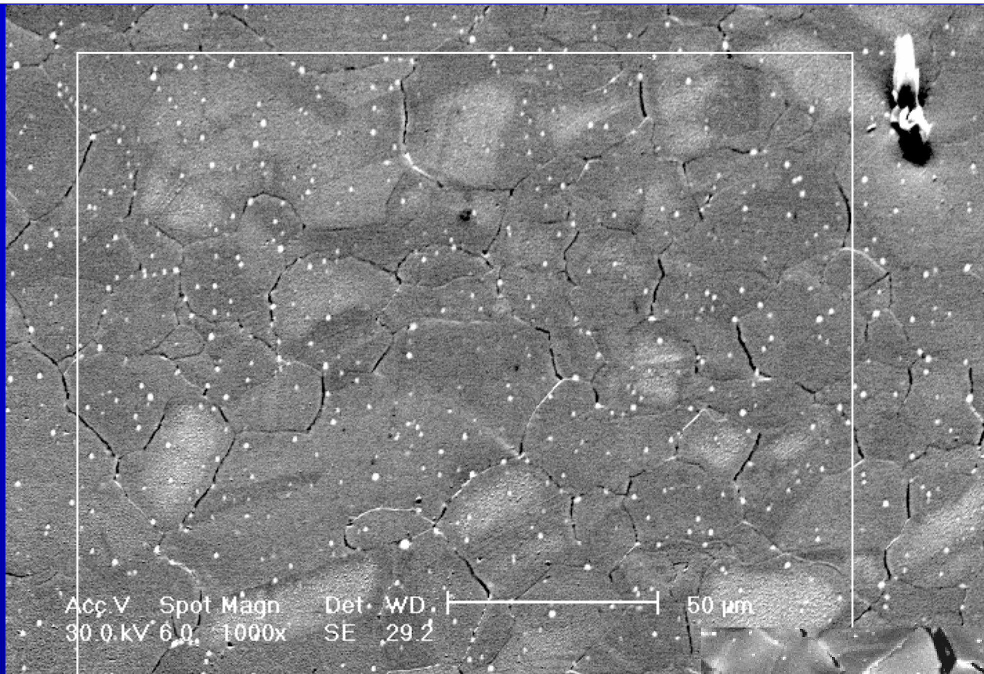
Sequential imaging and OIM during *in-situ* creep deformation

Much care is needed to identify crack vs. ledge. SE imaging are more useful than IQ maps



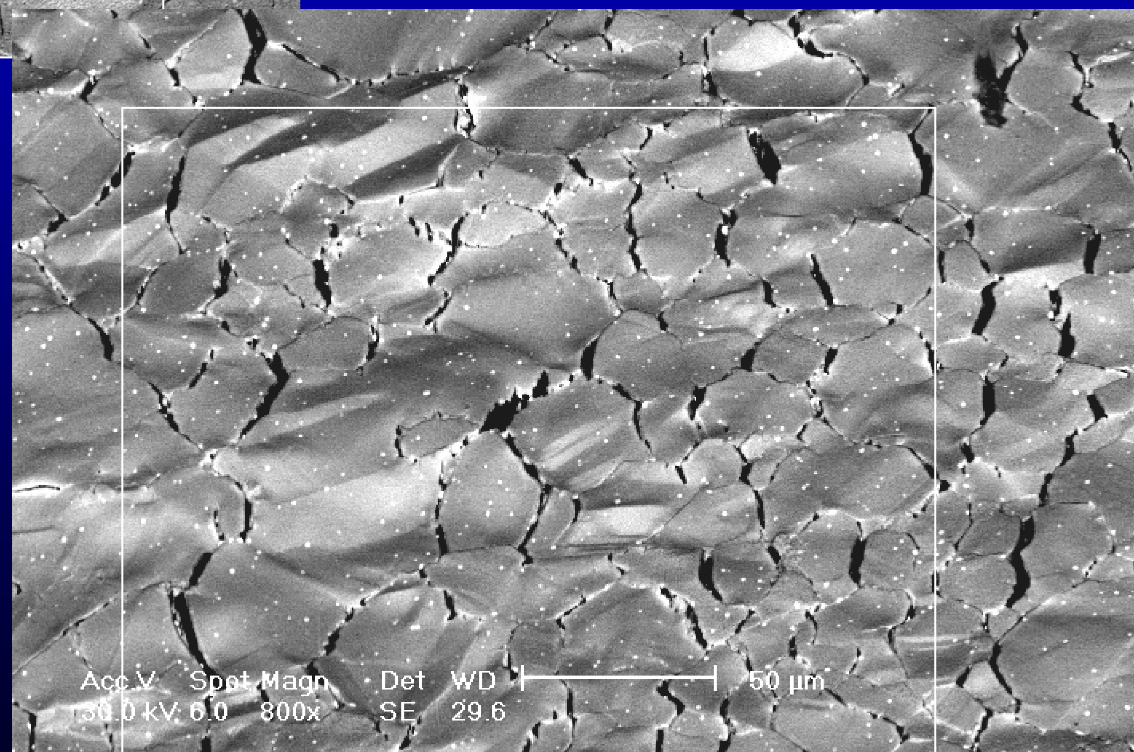


(a)

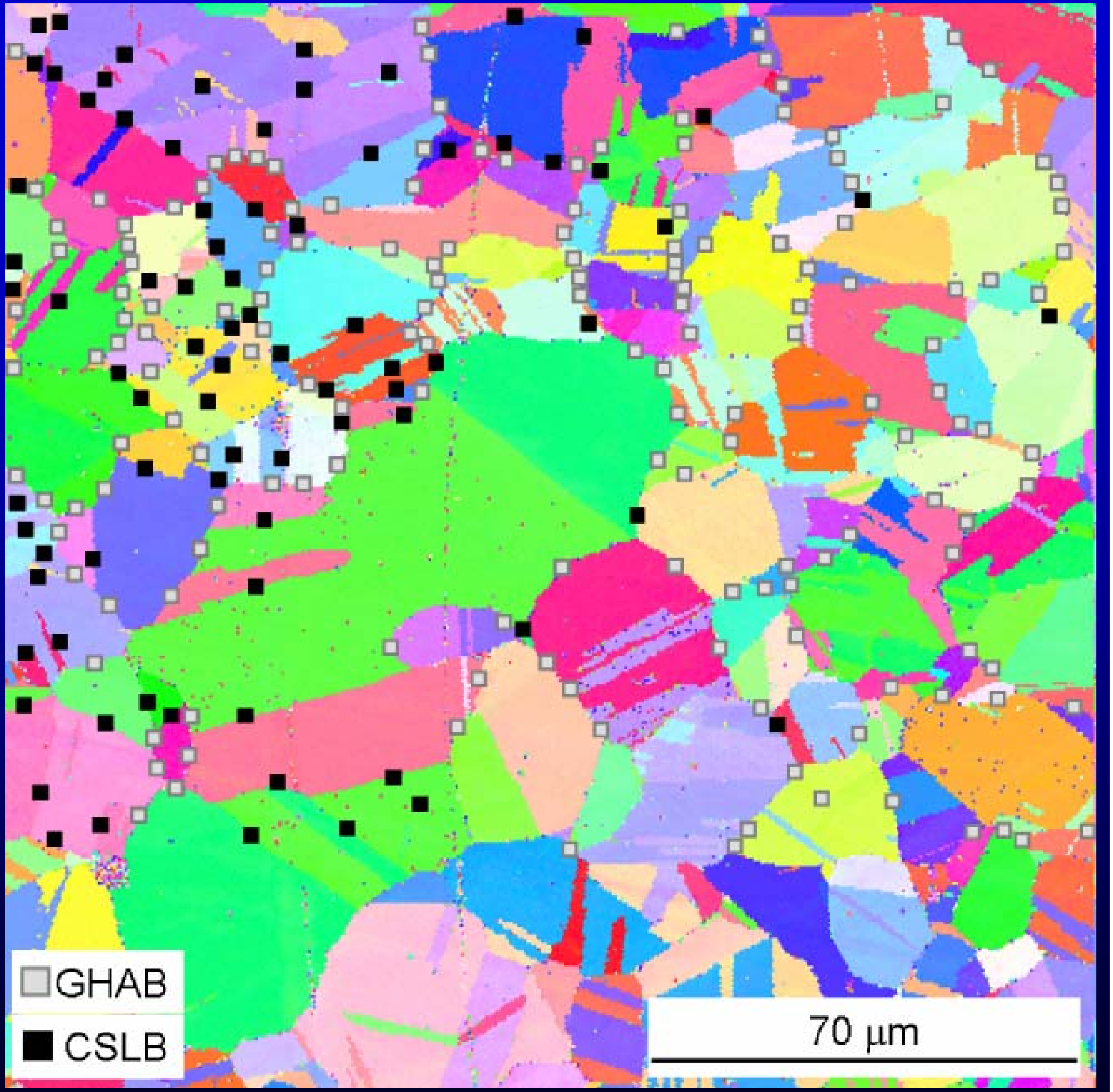


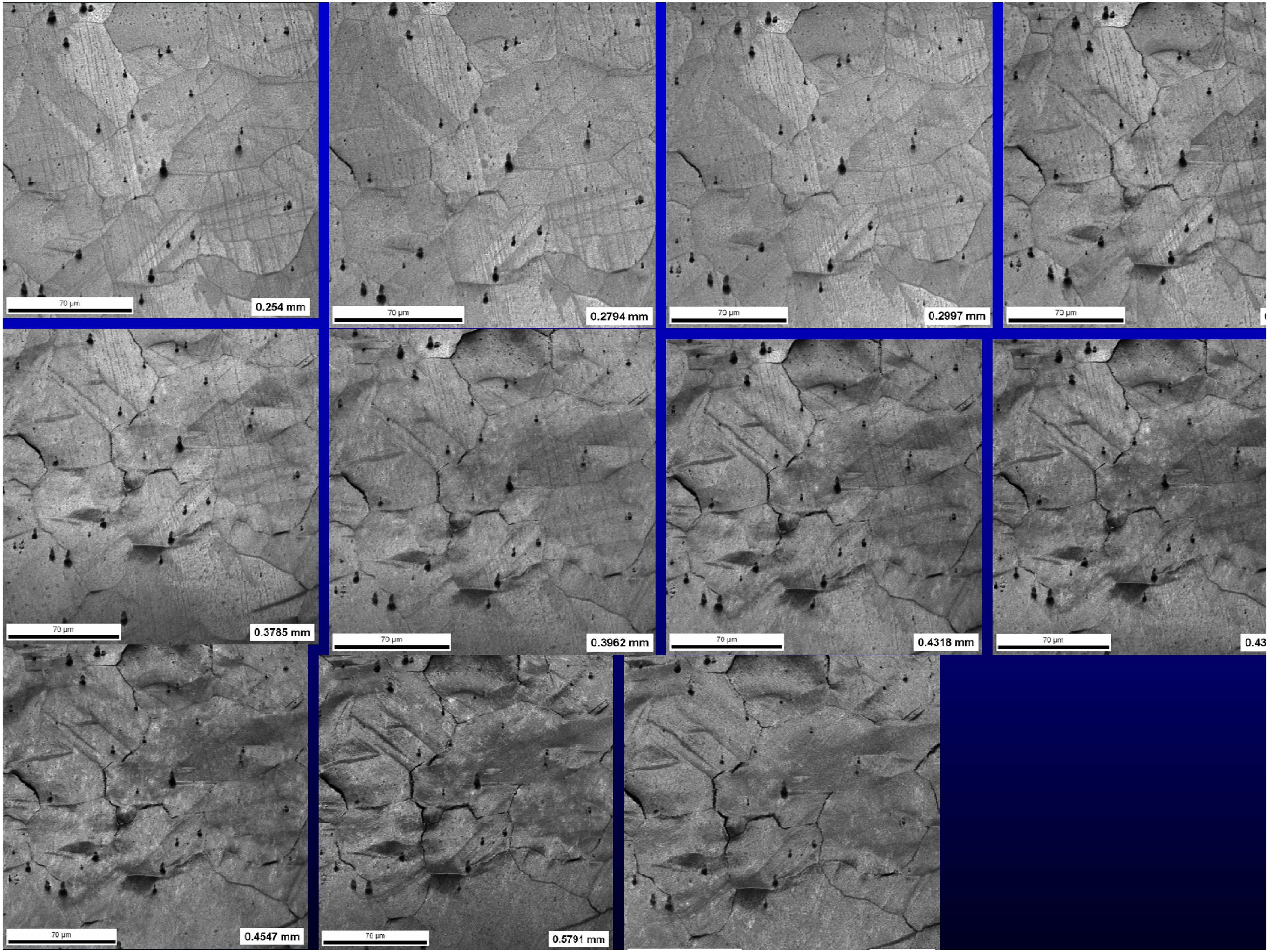
SE SEM micrographs for a 25% cold rolled specimen deformed at T=760°C and σ=220MPa after (a) 0.117mm displacement, (b) more than 1.039mm displacement creep. The area within the boxes was characterized using EBSD. The loading axis was horizontal.

(b)



EBSD inverse pole figure orientation map for a 25% cold rolled specimen prior to creep testing at T=760C and σ=220MPa. The GHABs and CSLBs that were analyzed are marked. The loading axis was horizontal.





0.254 mm
0.2794 mm
0.2997 mm
0.3785 mm
0.3962 mm
0.4318 mm
0.4547 mm
0.5791 mm