Update on PNNL SCC Growth Rate Testing on Alloy 690

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NRC Project Manager

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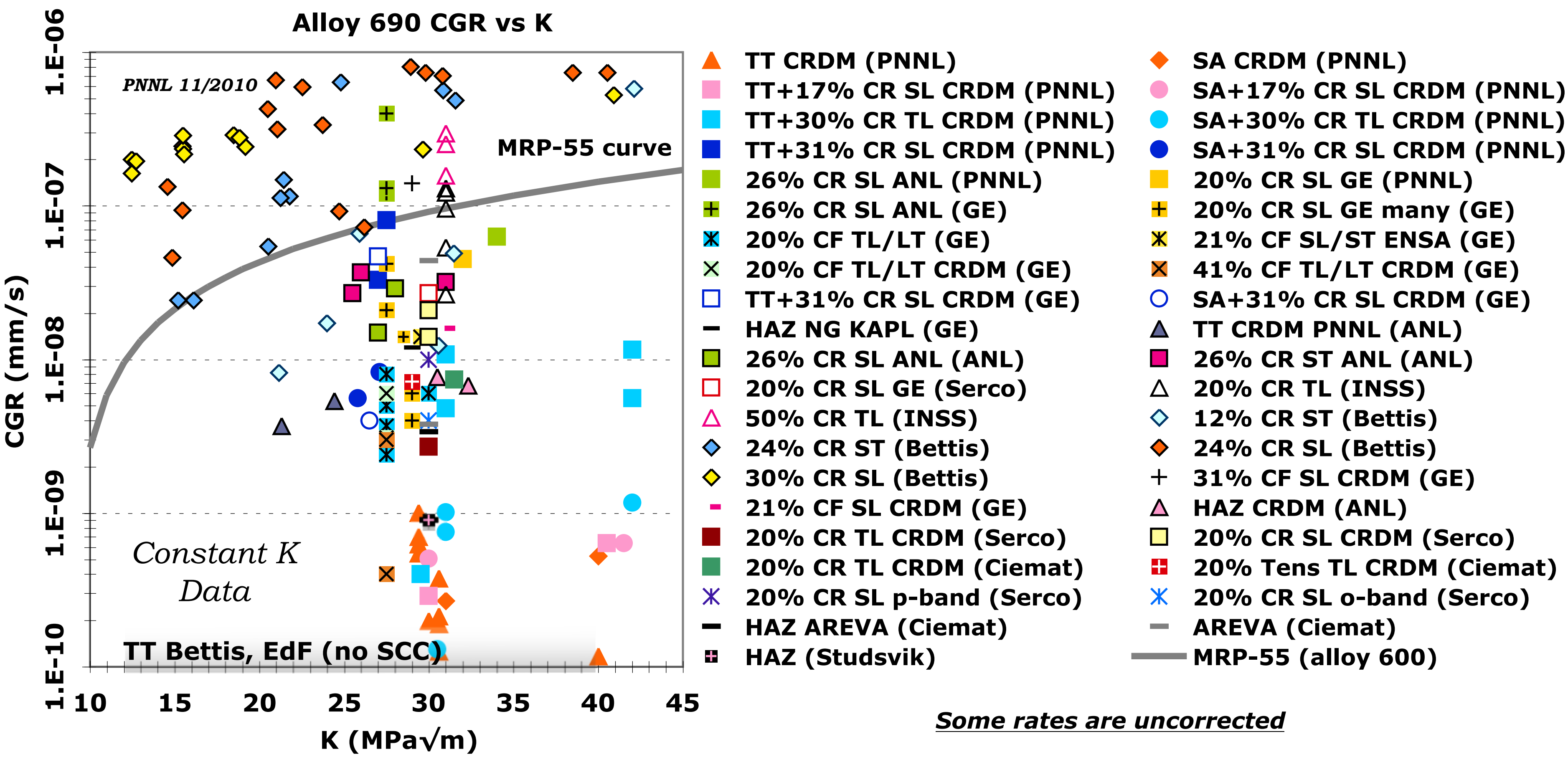
Summary of Alloy 690 Measurements of SCC Growth Rates – All Data



Some rates are uncorrected

Full spectrum of measured SCC growth rates illustrating significant effect of 1D cold work, however initial Bettis results remain at upper end of data with extremely high growth rates at lower K values.

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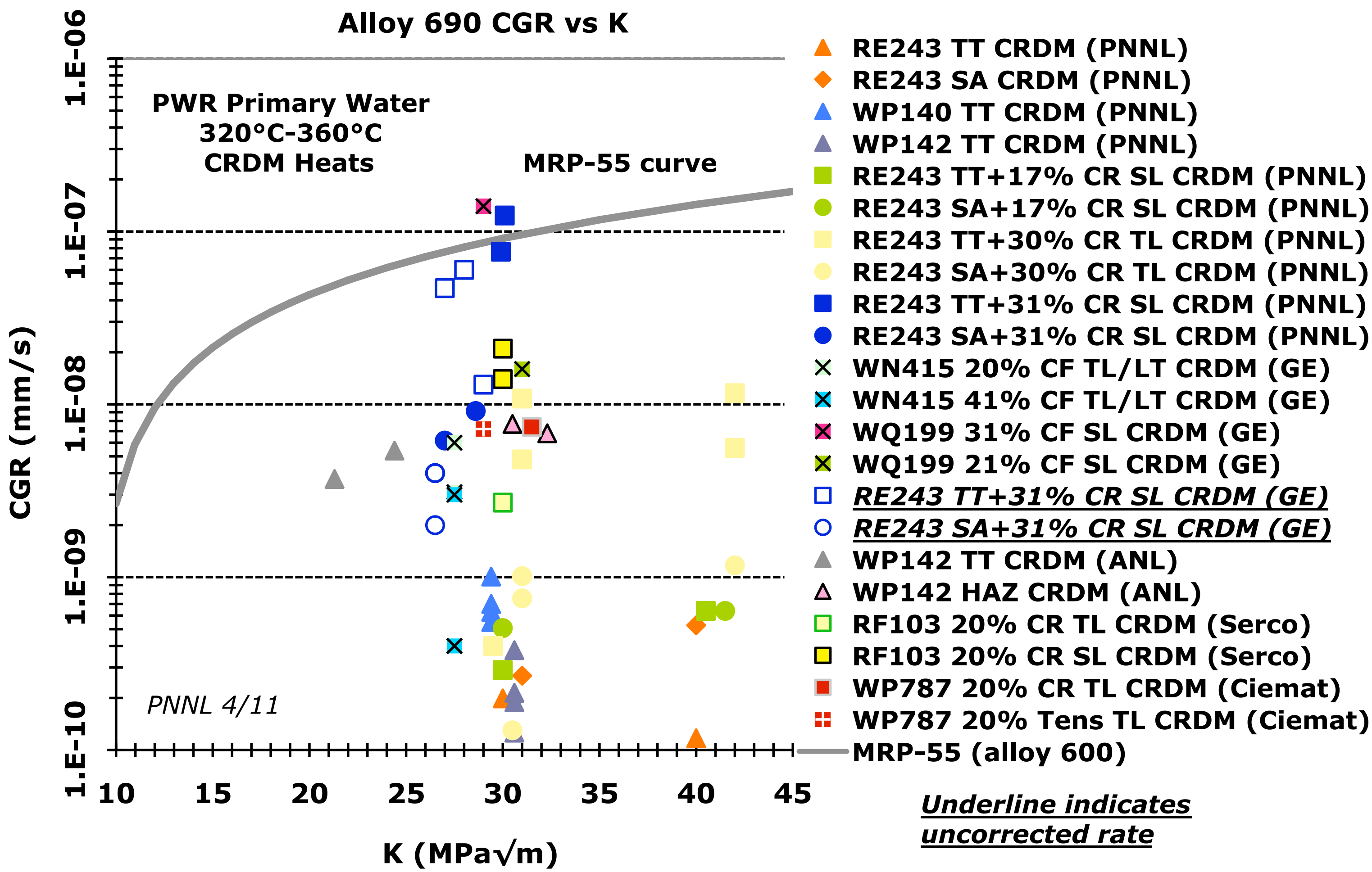
Measured SCC Growth Rates for Alloy 690 CRDM Tubing Materials



The number of SCC growth tests on cold-worked CRDM alloy 690 heats has increased significantly over the last 3 years. High levels of CW can promote moderate-to-high propagation rates.

PNNL Alloy 690 Testing Summary

- ***Heat-to-Heat Comparisons for CRDM Tubing***
  - *Three as-received TT heats: RE243, WP140 and 142*
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 CRDM Tubing***
  - *Single heat of alloy 690 CRDM tubing: heat RE243*
  - *As-received TT (high density of GB Cr carbides + Cr depletion) versus solution-annealed (no GB Cr carbides or Cr depletion)*
  - ***Cold rolling effects: 0%, 17% (S-L), 30% (T-L), 31% (S-L)***
  - *Post-cold rolling recovery anneal to modify dislocation structure keeping permanent damage: recently completed for 31%CR alloy 690TT + 700°C/1 hr/AC (S-L)*
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 Plate Materials***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - *Ongoing for 22%CR (S-L) and 30%CF (S-L/S-T) ENSA heat.*

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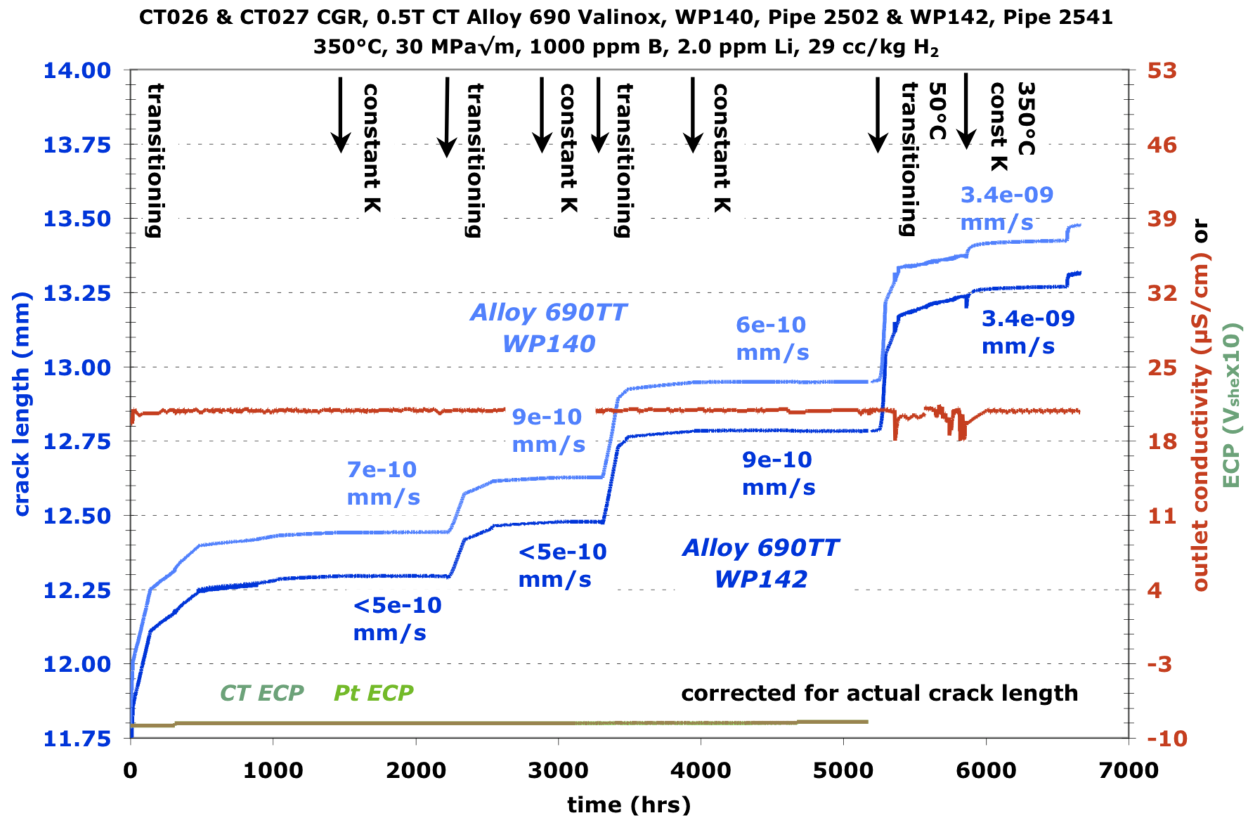
PNNL Crack Growth Rate Testing

- *Seven recirculating autoclave systems*
- *DCPD in-situ crack length measurement with <2 µm peak-to-peak noise*
- *Simulated PWR primary water (1000 ppm B, 2 ppm LiOH)*
- *Most testing at 360°C with H_2 at Ni-NiO line*
- *Focus on establishing constant K response after various SCC transitioning steps*
- *Growth rates adjusted for post-test crack length observations*



Example of testing approach for two as-received CRDM heats: constant K typically evaluated several times in different microstructural regions and often at different K levels during long-term tests.

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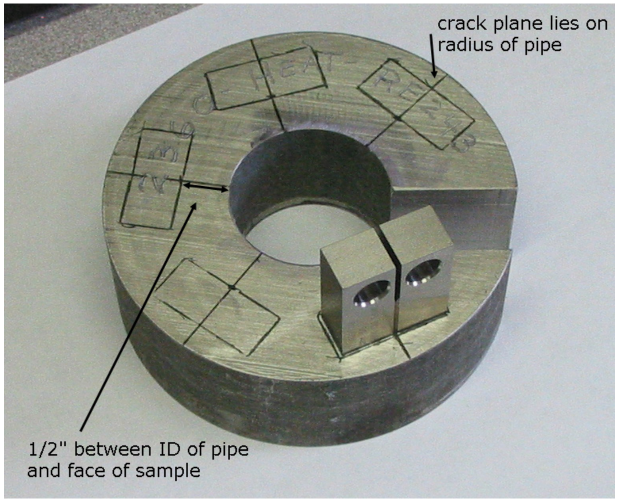
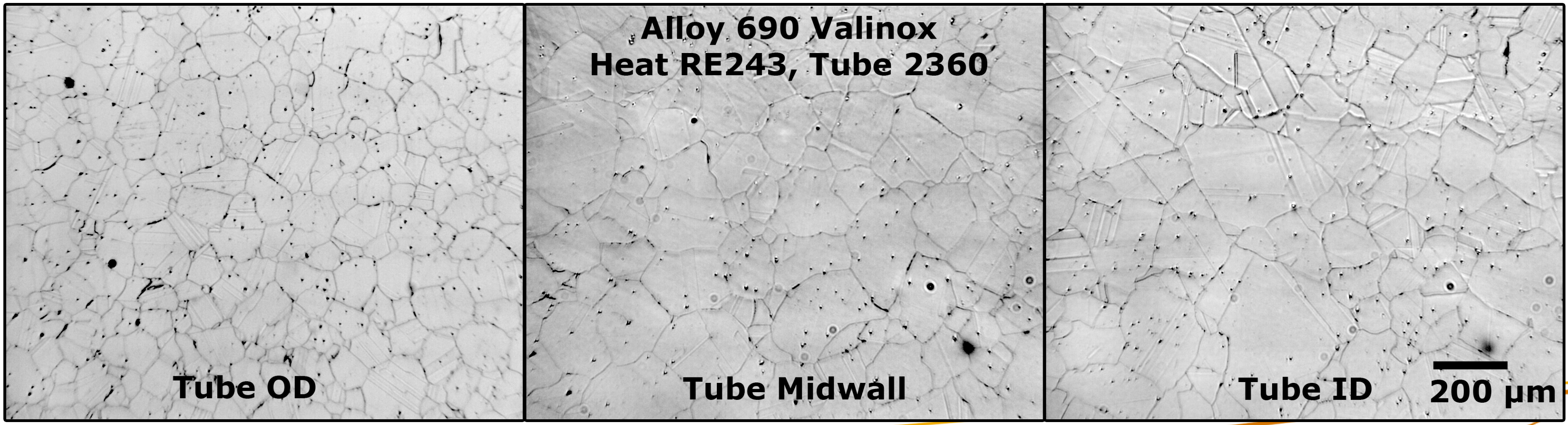
As-Received, Alloy 690TT CRDM Tubing

- *Extruded tubing provided by Valinox in a thermally treated condition (720°C/ 10.5 hr)*
- *Equiaxed grain dimensions (70-120 µm) at midwall and ID, smaller very near tube OD*
- *High density of nearly continuous, grain boundary carbides*
- *Low density of matrix TiN particles*
- *No banded structures*



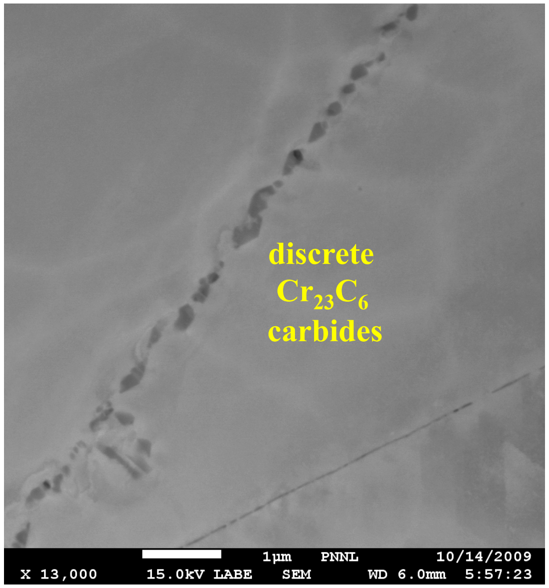


Microstructure, Hardness and Crack-Growth Rates for CR CRDM Alloy 690TT Heat RE243

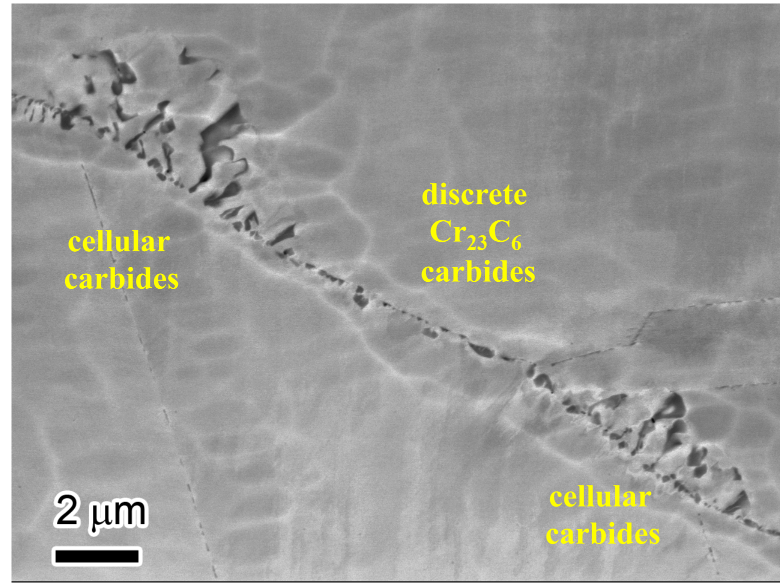
CRDM Alloy 690 Material	Microstructure	Test Plane Hardness	PNNL CGRs
TT	Nearly continuous GB carbides, isolated GB and matrix TiN, equiaxed grains	175 kg/mm^2	<5x10^{-10} mm/s (325°)
TT+17%CR	Nearly continuous GB carbides, isolated GB and matrix TiN, slightly elongated grains, high dislocation density	250 kg/mm^2	~2.5x10^{-9} mm/s (S-L, 360°C)
TT+30%CR	Nearly continuous GB carbides, isolated GB and matrix TiN; elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides	300 kg/mm^2	~1x10^{-8} mm/s (T-L, 350°C)
TT+31%CR	Nearly continuous GB carbides, isolated GB and matrix TiN; elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides	300 kg/mm^2	~1x10^{-7} mm/s (S-L, 360°C)

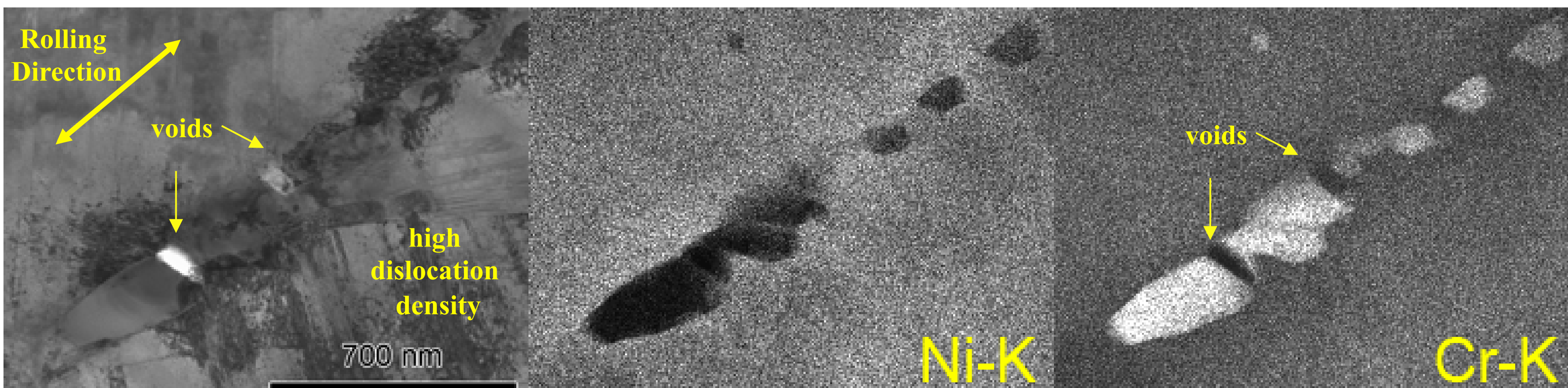
Cold-rolling increases SCC susceptibility of extruded alloy 690 tubing with "excellent" microstructure with growth rates reaching ~1x10^{-7} mm/s at high levels of deformation.

Cold Rolling Effects on Microstructure of Alloy 690TT CRDM



Nearly continuous $Cr_{23}C_6$ carbides along high-energy grain boundaries with regions of cellular ppt. Cold rolling produces voids between carbides and some cracked ppts.





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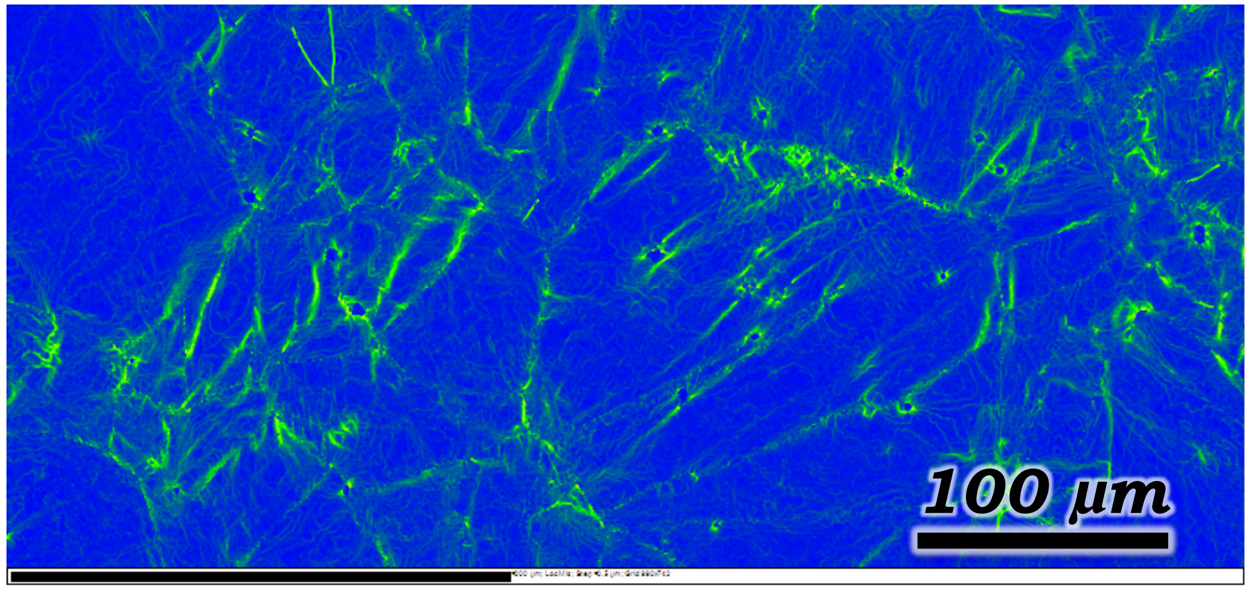
Effect of Cold Rolling on EBSD-Indicated Strain

Strain is linearly proportional to misorientation parameter

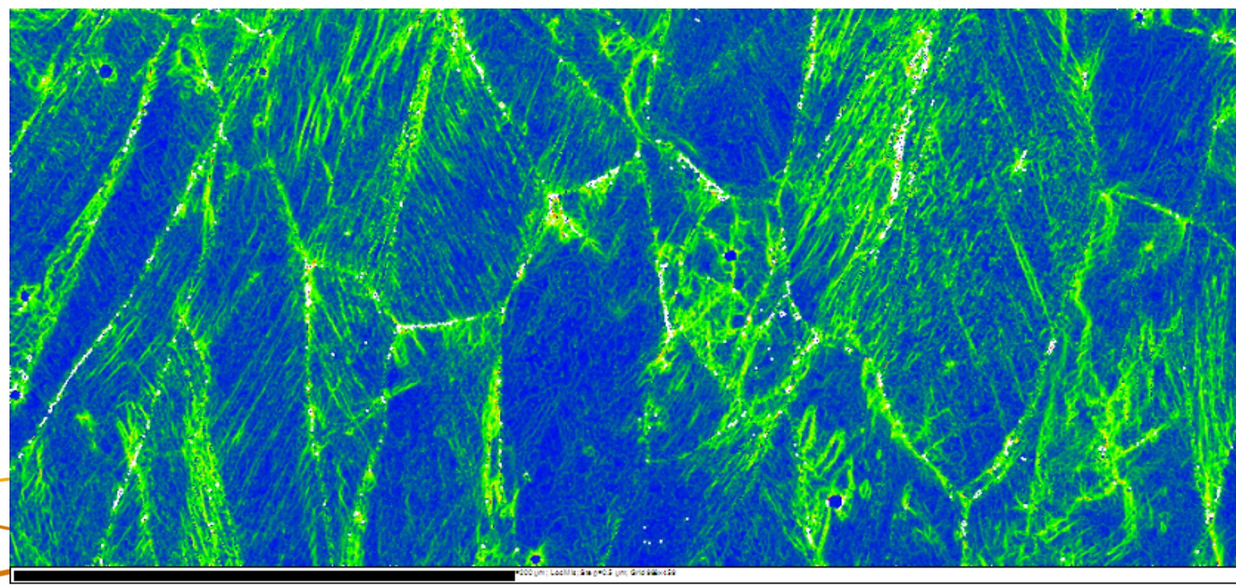
Misorientation represented by green intensity

17%CR
Alloy 690TT
250 kg/mm²

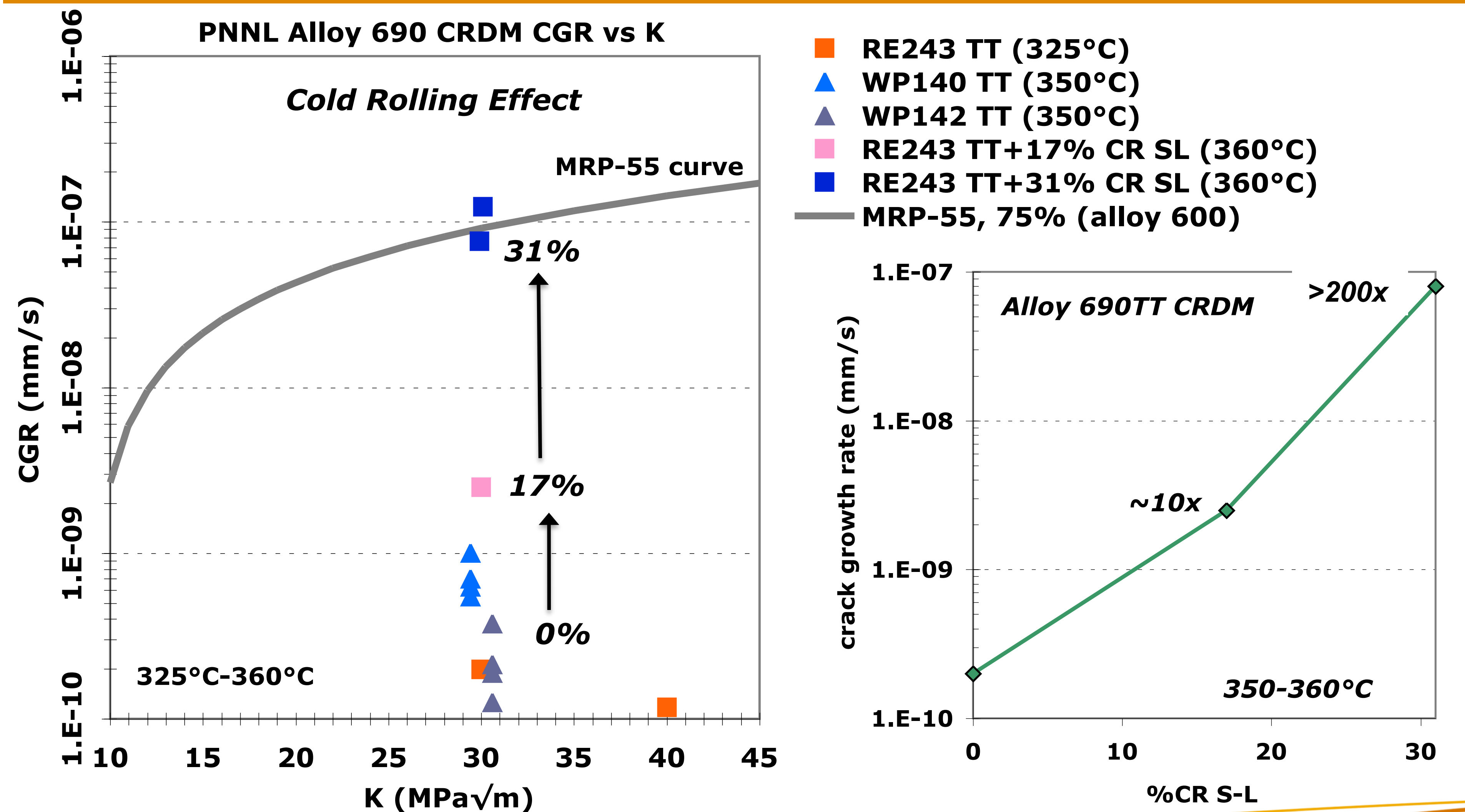


- *Significant difference in strains between 17% and 31%CR.*
- *Strains observed to develop first at GBs.*

31%CR
Alloy 690TT
300 kg/mm²

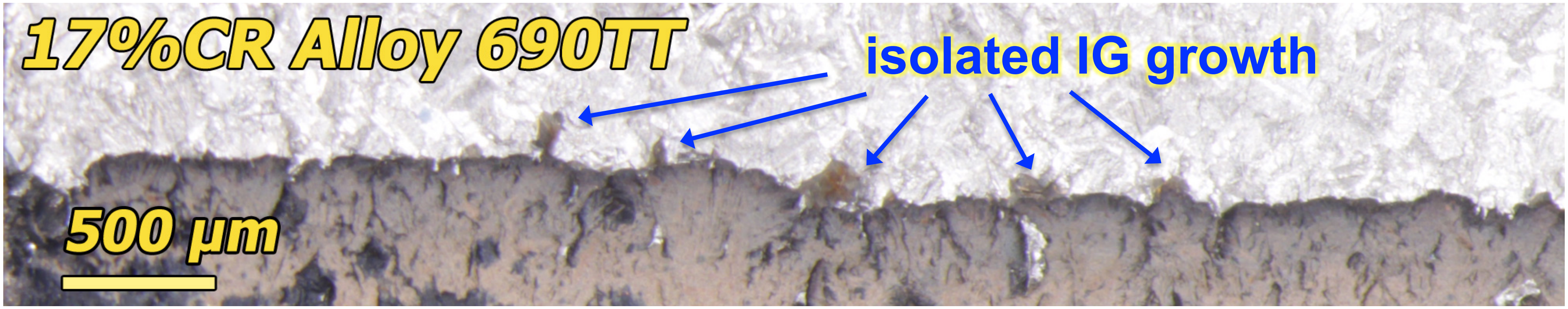


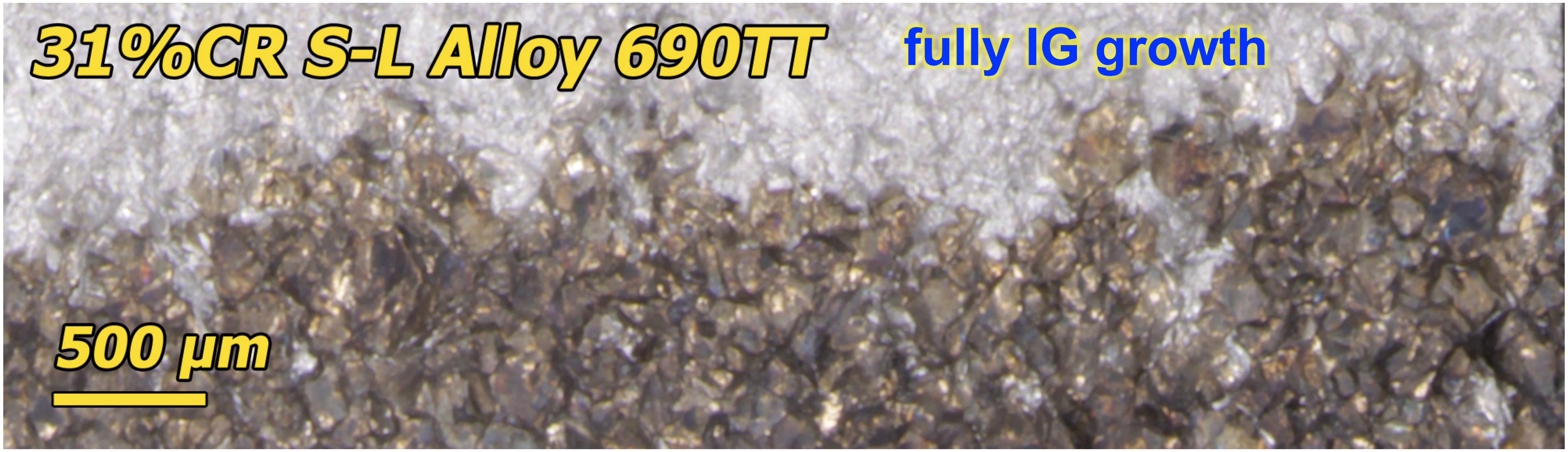
Cold Rolling Effect on Crack Growth Rate



Cold-rolling increases SCC susceptibility of extruded alloy 690 CRDM heat with "excellent" microstructure at high deformation levels.

Degree of Engagement in CR Specimens



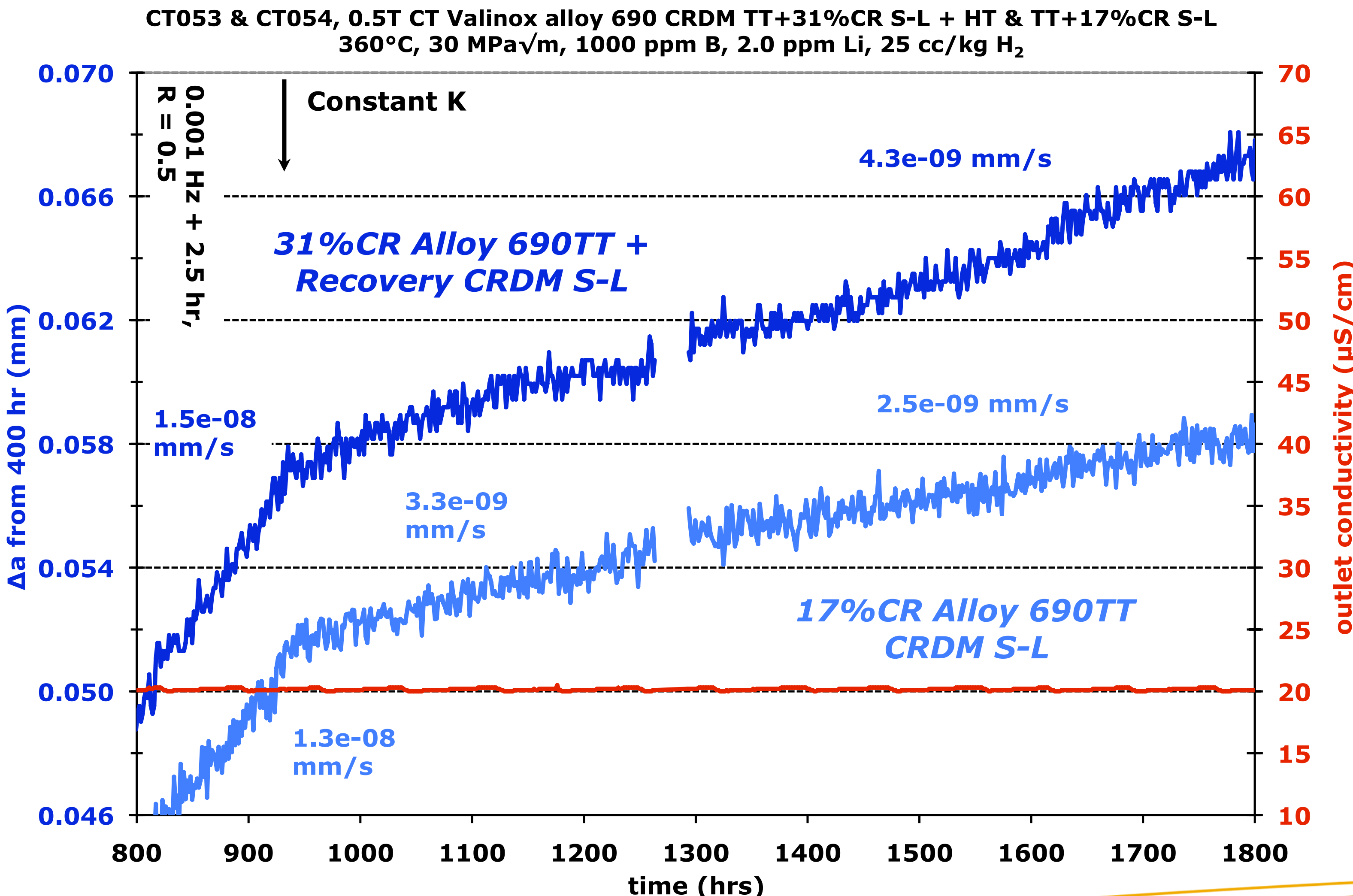


0%CR had no significant IG extension while 17%CR has a few isolated outcroppings of IG extension, and 31%CR has a fully IG crack front.

PNNL Alloy 690 Testing Summary

- ***Heat-to-Heat Comparisons for CRDM Tubing***
  - *Three as-received TT heats: RE243, WP140 and 142*
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 CRDM Tubing***
  - *Single heat of alloy 690 CRDM tubing: heat RE243*
  - *As-received TT (high density of GB Cr carbides + Cr depletion) versus solution-annealed (no GB Cr carbides or Cr depletion)*
  - *Cold rolling effects: 0%, 17% (S-L), 30% (T-L), 31% (S-L)*
  - ***Post-cold rolling recovery anneal to modify dislocation structure keeping permanent damage: recently completed for 31%CR alloy 690TT + 700°C/1 hr/AC (S-L)***
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 Plate Materials***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - *Ongoing for 22%CR (S-L) and 30%CF (S-L/S-T) ENSA heat.*

Effect of a Recovery Anneal on Crack Growth Rate for 31%CR Alloy 690TT



Recovery anneal for 31%CR alloy 690TT reduced measured SCC growth rates from ~1x10^{-7} mm/s, becomes similar to 17%CR response.

Microstructure, Hardness and Crack-Growth Rates for CR CRDM Alloy 690TT

CRDM Alloy 690 Material	Microstructure	Test Plane Hardness	PNNL CGRs
TT	Nearly continuous GB carbides, isolated GB and matrix TiN, equiaxed grains	175 kg/mm^2	<$5x10^{-10}$ mm/s (325°)
TT+17%CR	Nearly continuous GB carbides, isolated GB and matrix TiN, slightly elongated grains, high dislocation density	250 kg/mm^2	~$2.5x10^{-9}$ mm/s (S-L, 360°C)
TT+31%CR	***Nearly continuous GB carbides, isolated GB and matrix TiN; elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides***	***300 kg/mm^2***	***~$1x10^{-7}$ mm/s (S-L, 360°C)***
TT+31%CR + 700°C/1 hr/AC	***No significant change from TT+31%CR, some alteration of dislocation microstructure***	***270 kg/mm^2***	***~$4x10^{-9}$ mm/s (S-L, 360°C)***

Recovery heat treatment selected to modify dislocation structure and reduce hardness, but have no effect on permanent grain boundary damage (voids and cracked carbides) and general microstructure. Higher temperatures or longer times at 700^{o}C promoted local recrystallization.

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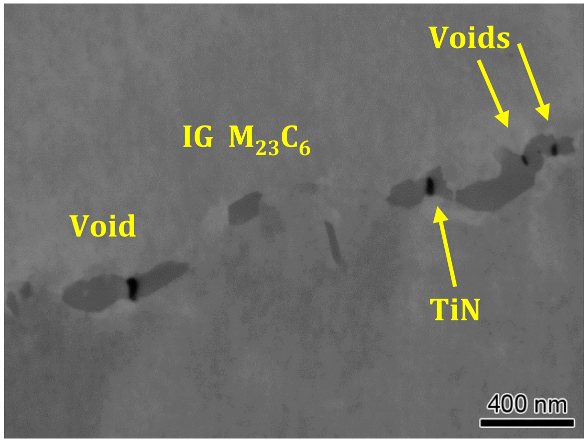
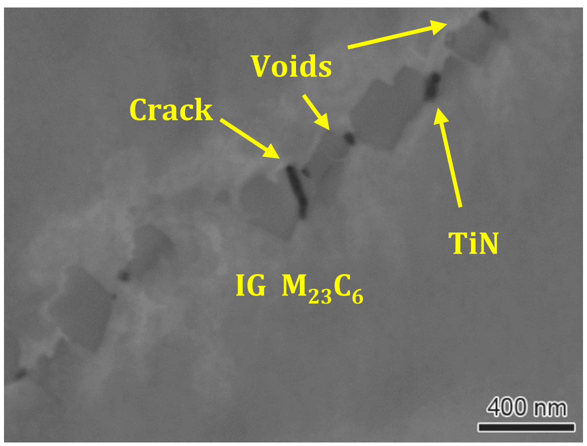
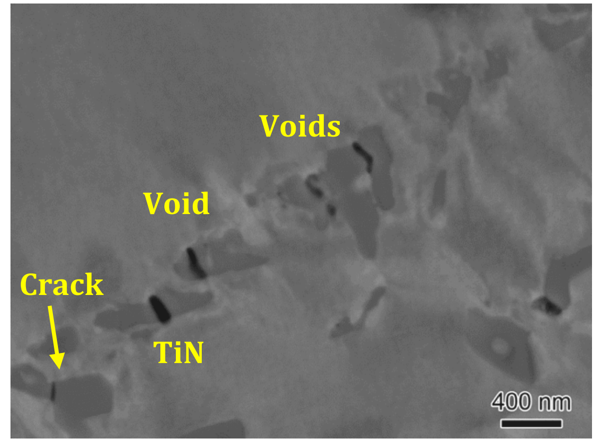
Effect of Recovery Anneal on the Permanent GB Damage Microstructures

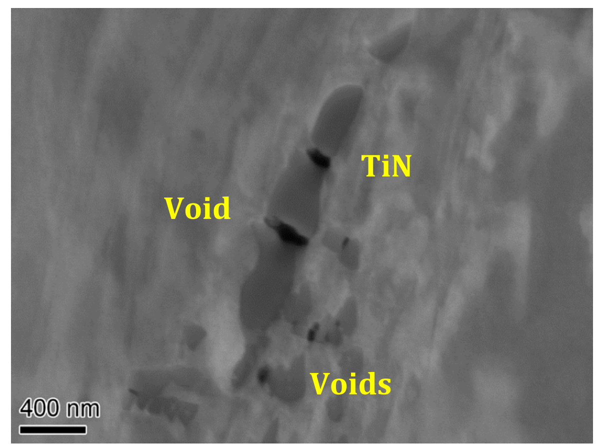
31%CR alloy 690TT CRDM





31%CR alloy 690TT + Recovery Anneal





A moderate density of GB voids and some cracked carbides present in 30-31%CR alloy 690TT material and ***similar density remains after the post-CR 700°C recovery anneal.***

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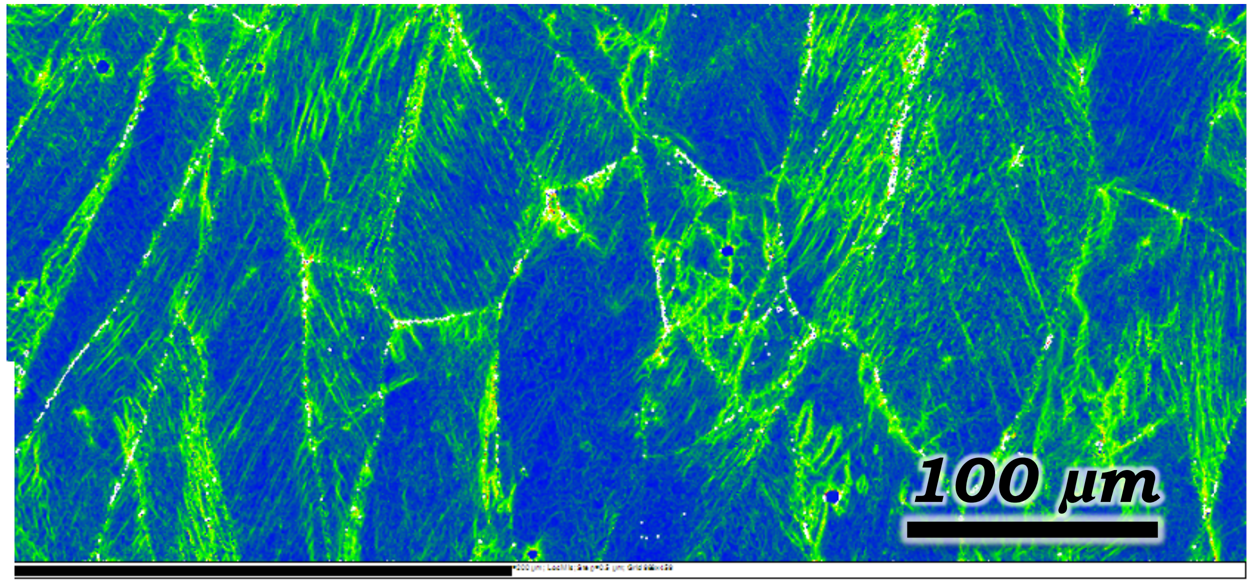
Effect of Recovery Treatment on EBSD-Indicated Strain

Strain is linearly proportional to misorientation parameter

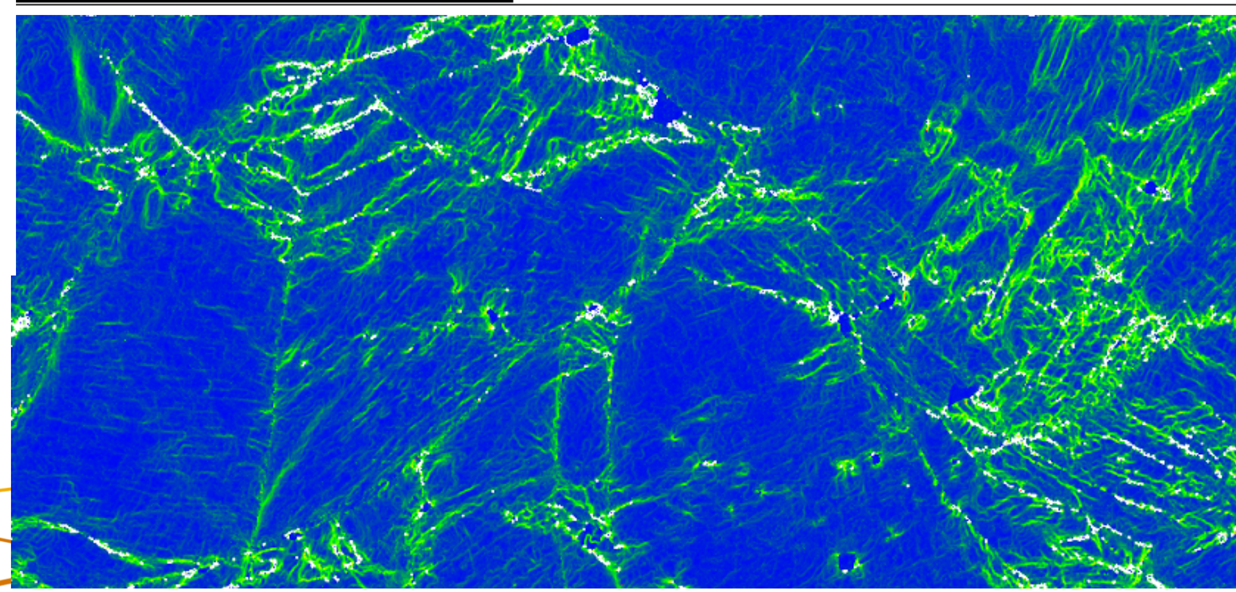
Misorientation represented by green intensity

31%CR
Alloy 690TT
300 kg/mm²

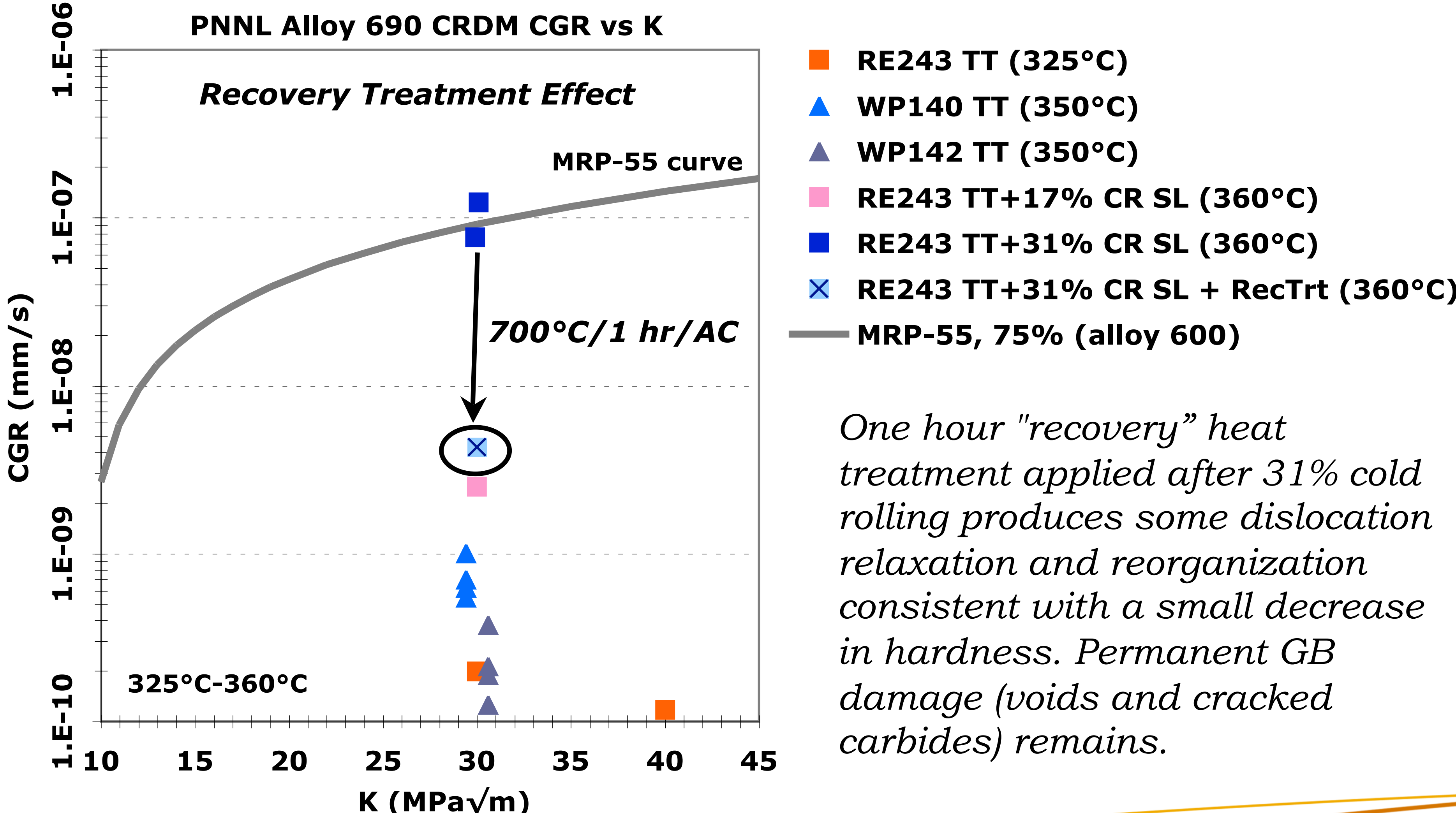


One hour "recovery" heat treatment was applied after 31%CR to produce some dislocation relaxation/reorganization. Permanent GB damage (voids and cracked carbides) remains.

31%CR
Alloy 690TT,
700°C/1 hr/AC
270 kg/mm²



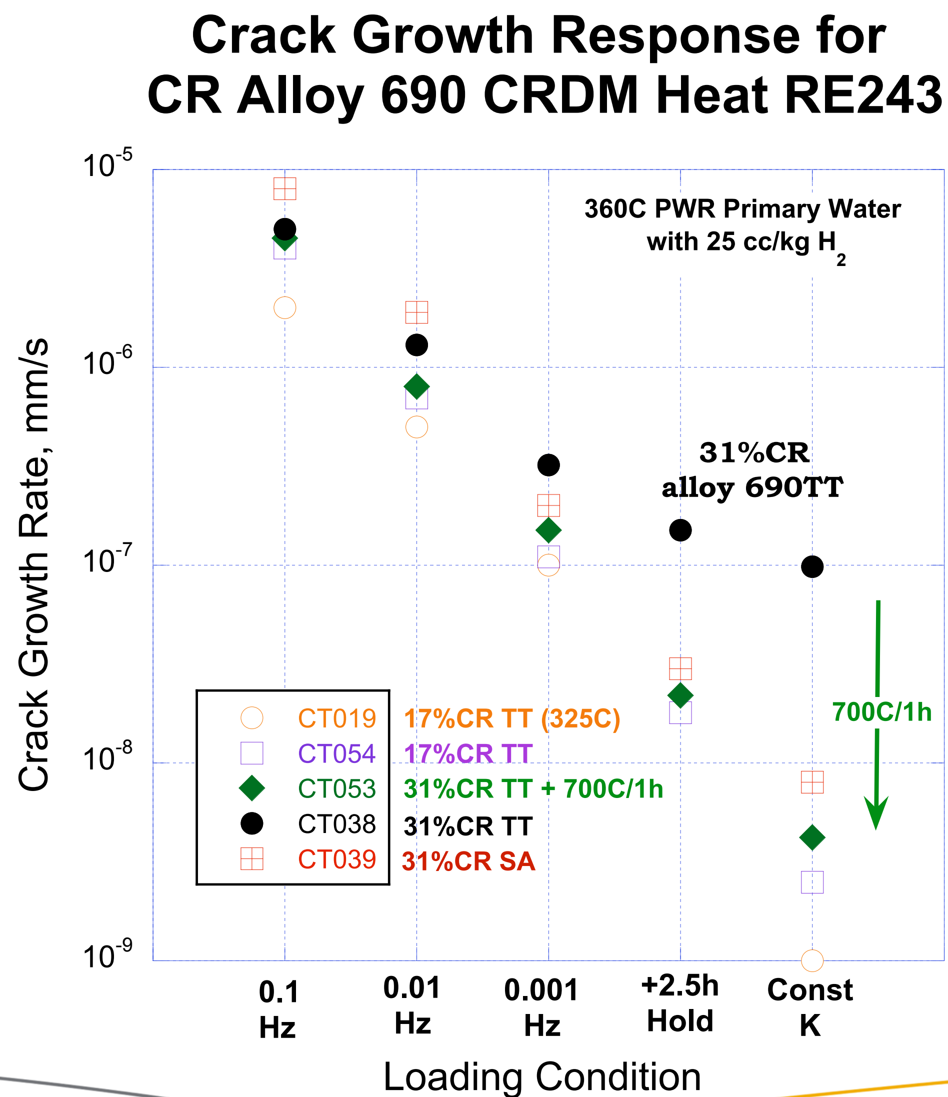
Effect of Recovery Heat Treatment on 31%CR Alloy 690TT Material



One hour "recovery" heat treatment applied after 31% cold rolling produces some dislocation relaxation and reorganization consistent with a small decrease in hardness. Permanent GB damage (voids and cracked carbides) remains.

700°C recovery heat treatment applied to 31%CR material results in a 20x lower crack growth rate. CGR is only slightly higher than 17% CR.

Effect of Recovery Heat Treatment on 31%CR Alloy 690TT Material



One hour "recovery" heat treatment applied after 31% cold rolling produces some dislocation relaxation and reorganization consistent with a small decrease in hardness. Permanent GB damage (voids and cracked carbides) remains.

700°C recovery heat treatment applied to 31%CR material results in a 20x lower SCC growth rate (can be seen during cycle + hold and constant K). Growth rate is similar to that for 17%CR material.

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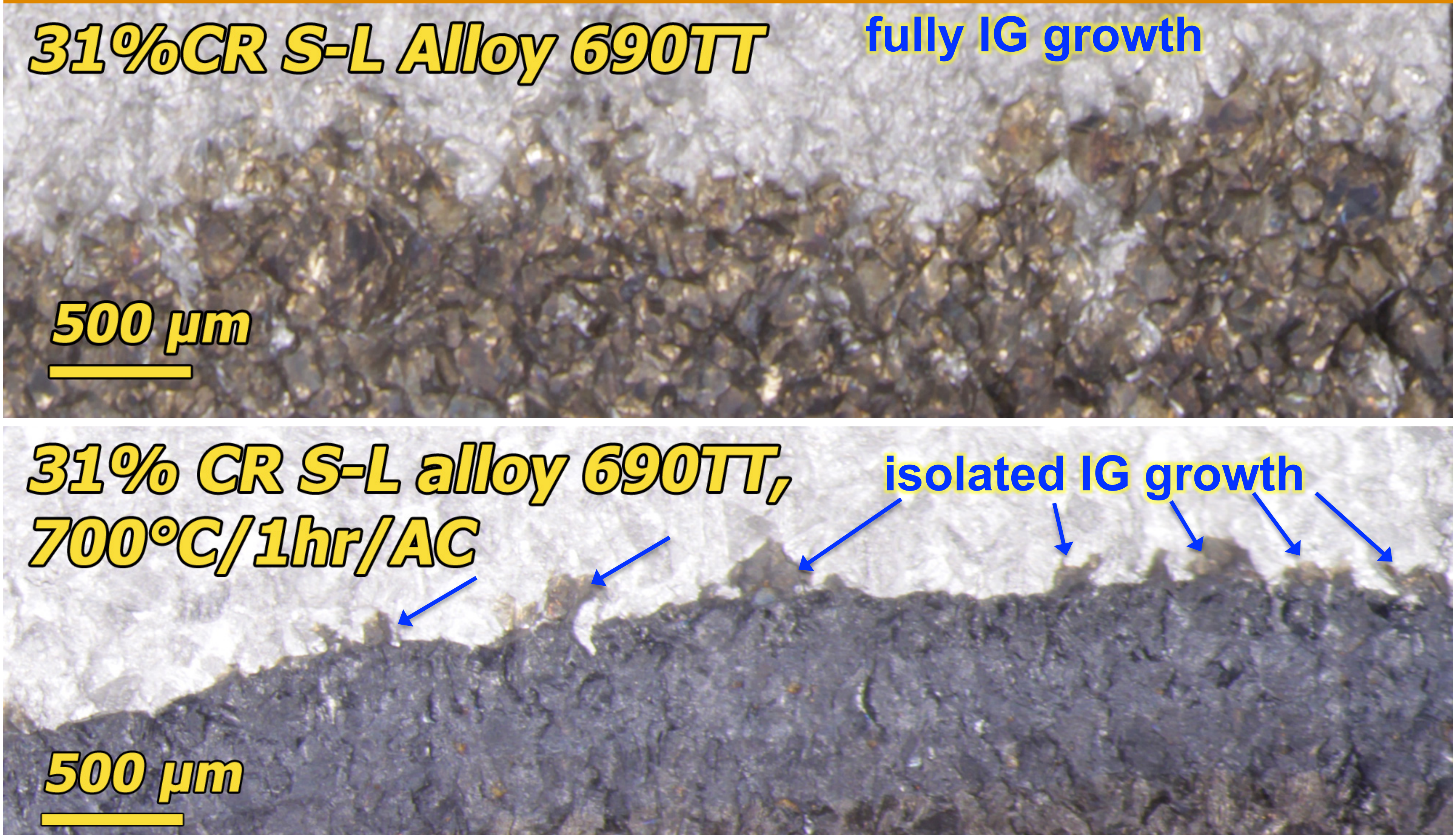
Effect of Recovery Heat Treatment



700°C/ 1 hr/ AC "recovery heat treatment" reduces IG engagement and CGR.

Microstructure, Hardness and Crack-Growth Rates for CR CRDM Alloy 690TT

CRDM Alloy 690 Material	*Microstructure*	*Test Plane Hardness*	*PNNL CGRs*
TT	Nearly continuous GB carbides, isolated GB and matrix TiN, equiaxed grains	175 kg/mm^2	<$5x10^{-10}$ mm/s (325°C)
TT+17%CR	Nearly continuous GB carbides, isolated GB and matrix TiN, slightly elongated grains, high dislocation density	250 kg/mm^2	~$2.5x10^{-9}$ mm/s (S-L, 360°C)
TT+31%CR	Nearly continuous GB carbides, isolated GB and matrix TiN; elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides	300 kg/mm^2	~$1x10^{-7}$ mm/s (S-L, 360°C)
TT+31%CR + 700°C/1 hr/AC	No significant change from TT+31%CR except for some alteration of dislocation microstructure	270 kg/mm^2	~$4x10^{-9}$ mm/s (S-L, 360°C)

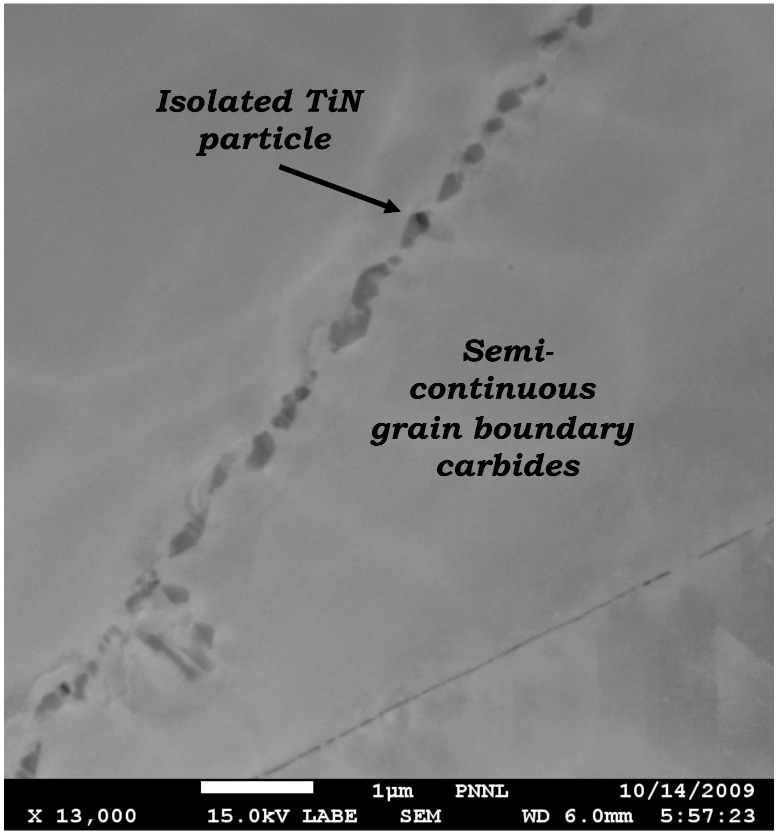
High levels of cold rolling increases SCC CGRs by ~200X in CRDM alloy 690TT material. Recovery heat treatment modifies dislocation structure significantly reduces IGSCC susceptibility in the 31%CR material, measured crack growth rate approaches 17%CR material.

PNNL Alloy 690 Testing Summary

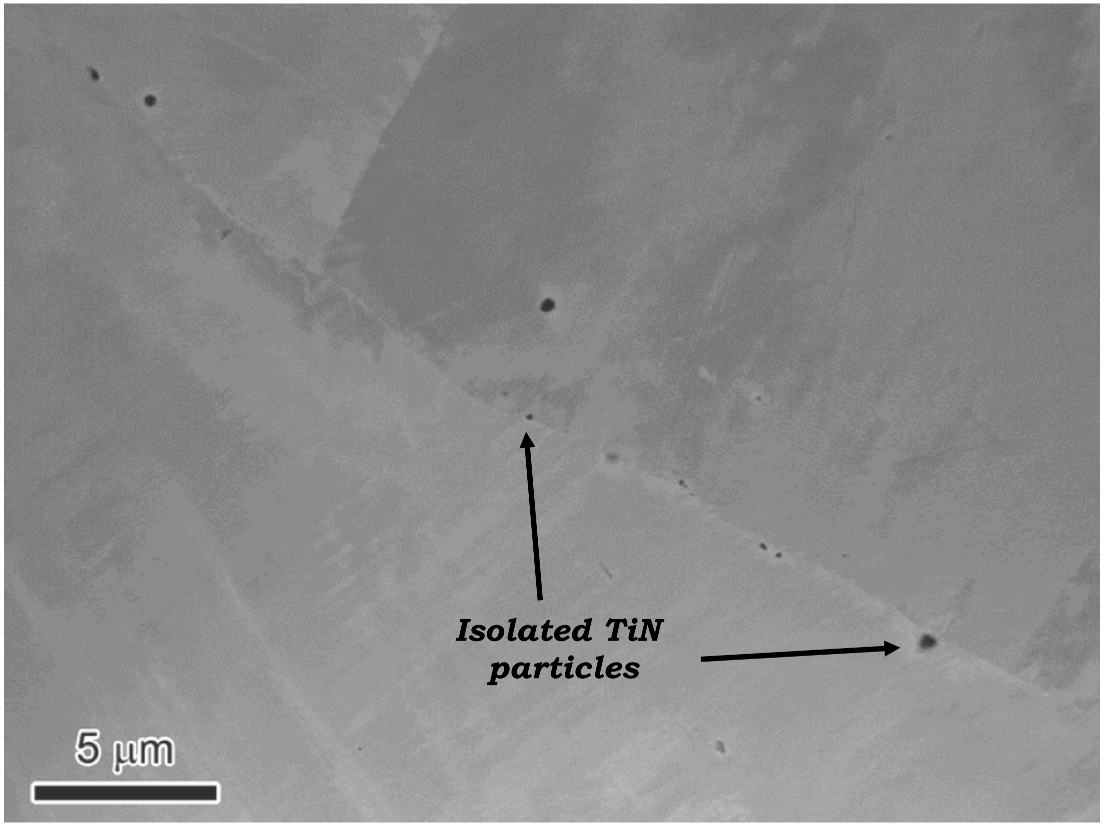
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 CRDM Tubing***
  - *Single heat of alloy 690 CRDM tubing: heat RE243*
  - ***As-received TT (high density of GB Cr carbides + Cr depletion) versus solution-annealed (no GB Cr carbides or Cr depletion)***
  - *Cold rolling effects: 0%, 17% (S-L), 30% (T-L), 31% (S-L)*
  - *Post-cold rolling recovery anneal to modify dislocation structure keeping permanent damage: recently completed for 31%CR alloy 690TT + 700°C/1 hr/AC (S-L)*
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 Plate Materials***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - *Ongoing for 22%CR (S-L) and 30%CF (S-L/S-T) ENSA heat.*

Grain Boundary Microstructures in Alloy 690 Thermally Treated (TT) versus Solution Annealed (SA)

Alloy 690TT CRDM



Alloy 690TT + SA



Solution anneal at 1100^{o}C and water quench removed nearly all grain boundary carbides, isolated TiN particles remain.

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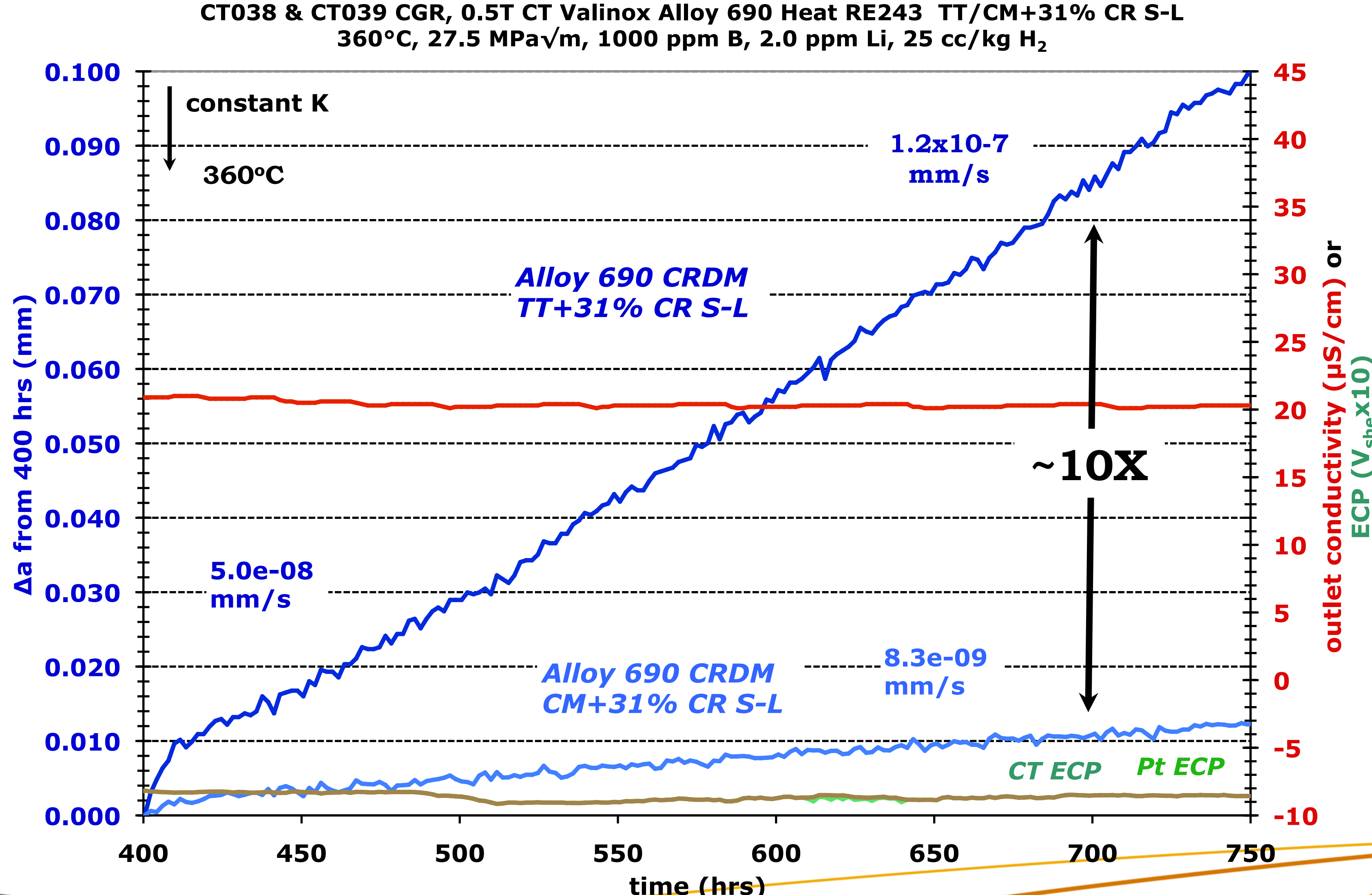
Alloy 690 CRDM Heat RE243 31%CR S-L Constant K



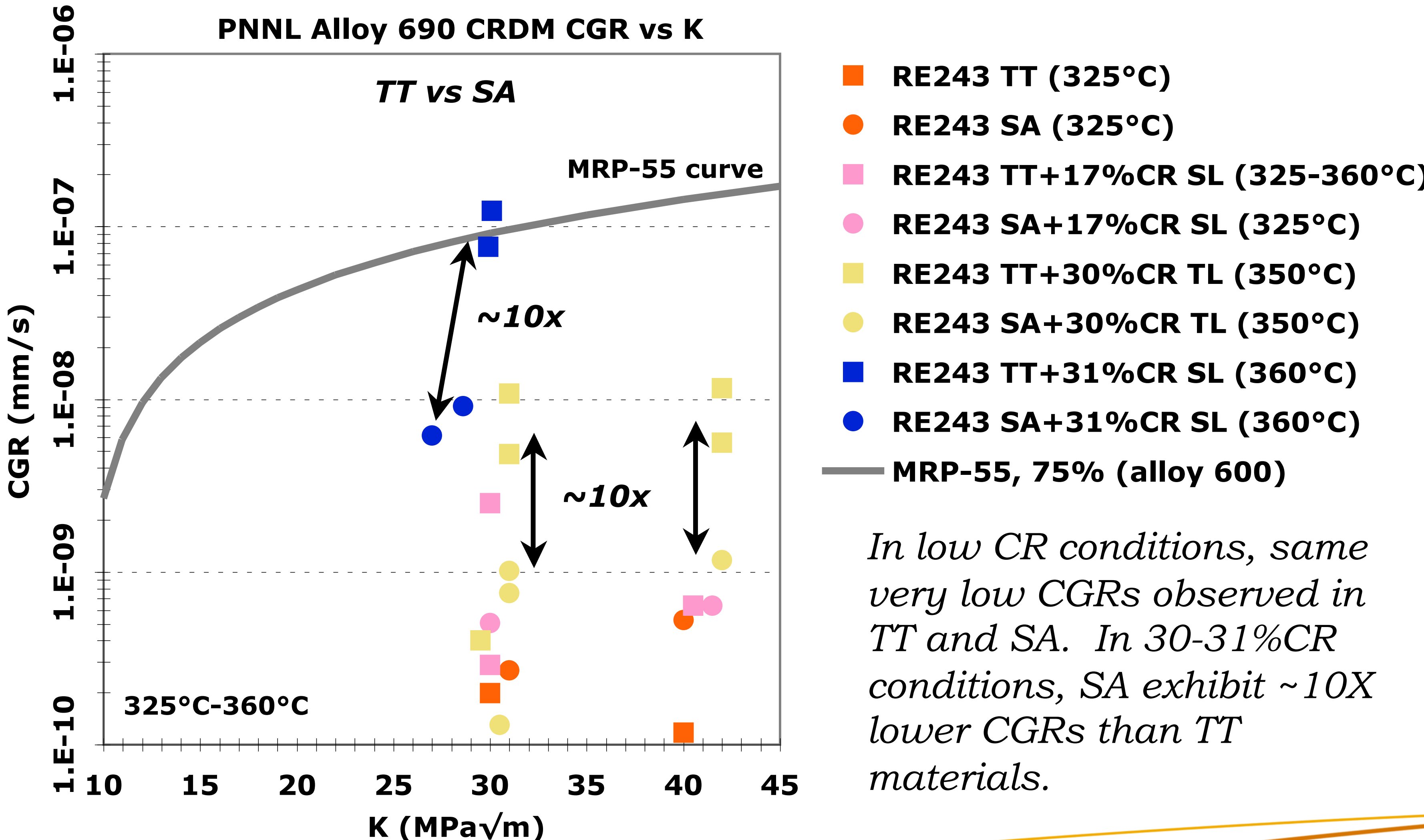
Higher crack-growth rate (~10X) in 31%CR alloy 690TT, reproduced at lower temps.

Microstructure, Hardness and Crack-Growth Rates for CR CRDM Alloy 690TT and 690SA

CRDM Alloy 690 Material	Microstructure	Test Plane Hardness	PNNL CGRs
TT	Nearly continuous GB carbides, isolated GB and matrix TiN	175 kg/mm^2	≤5x10^{-10} mm/s (325°C)
SA 1100°C/1hr/WQ	Isolated GB and matrix TiN, slightly elongated grains, high dislocation density	165 kg/mm^2	≤5x10^{-10} mm/s (325°C)
TT+17%CR	Like TT, but with slightly elongated grains, high dislocation density	250 kg/mm^2	~2.5x10^{-9} mm/s (S-L, 360°C)
SA+17%CR	Like SA, but with slightly elongated grains, high dislocation density	240 kg/mm^2	~5x10^{-10} mm/s (S-L, 325°C)
TT+30%CR	Like TT, but with elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides	300 kg/mm^2	~1x10^{-8} mm/s (T-L, 350°C)
SA+30%CR	Like SA but with, elongated grains, very high dislocation density, few cracked TiN	290 kg/mm^2	~1x10^{-9} mm/s (T-L, 350°C)
TT+31%CR	Same as TT+30%CR	300 kg/mm^2	~1x10^{-7} mm/s (S-L, 360°C)
SA+31%CR	Same as SA+30%CR	290 kg/mm^2	~8x10^{-9} mm/s (S-L, 360°C)

High levels of unidirectional cold rolling increases SCC CGRs by ~200X in CRDM alloy 690TT material. Solution anneal prior to cold rolling reduces the influence of cold rolling on crack growth rates and IG engagement.

SCC Crack-Growth Rates for Alloy 690SA versus As-Received Alloy 690TT Materials



In low CR conditions, same very low CGRs observed in TT and SA. In 30-31%CR conditions, SA exhibit ~10X lower CGRs than TT materials.

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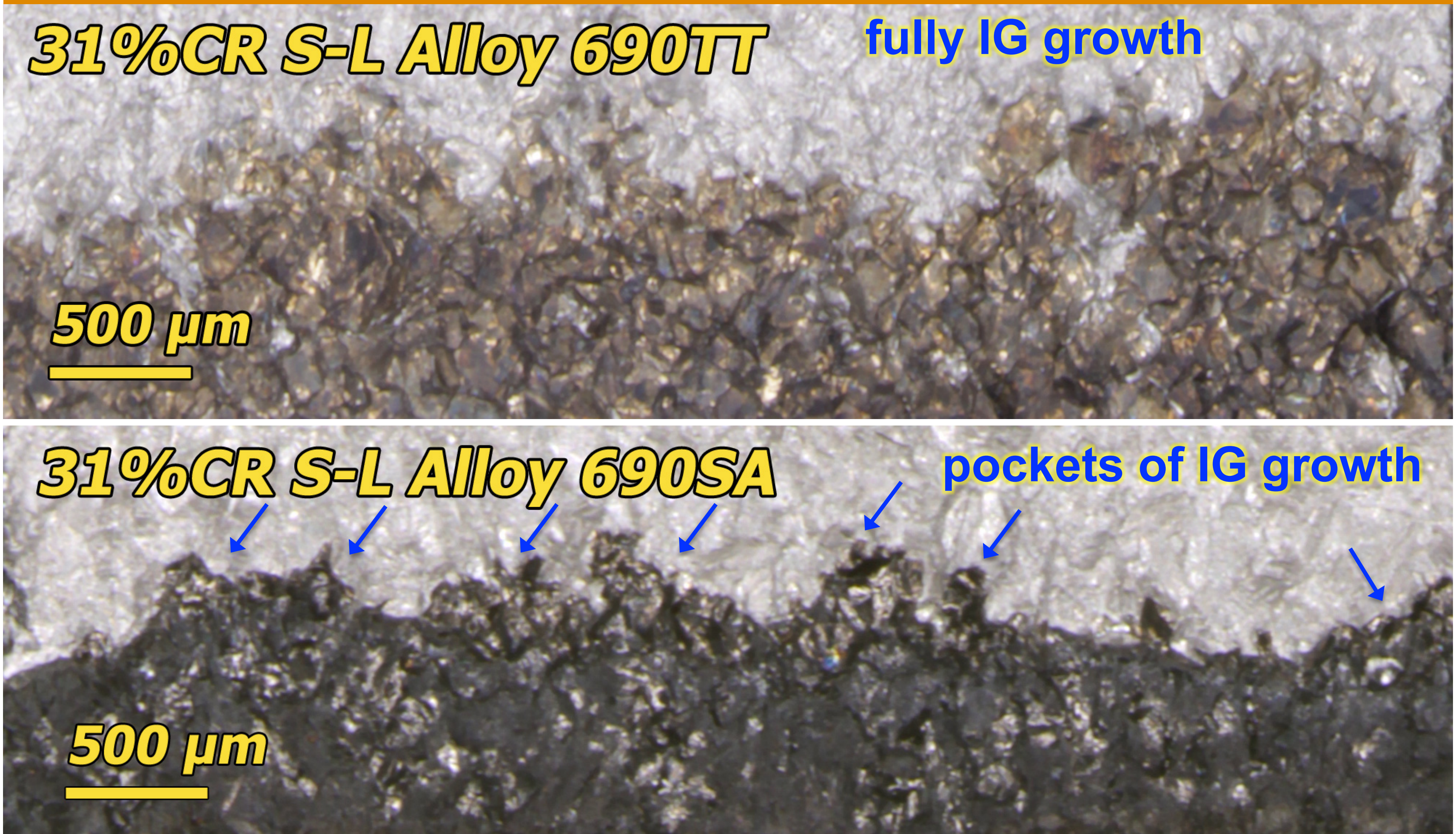
Thermally Treated vs Solution Annealed



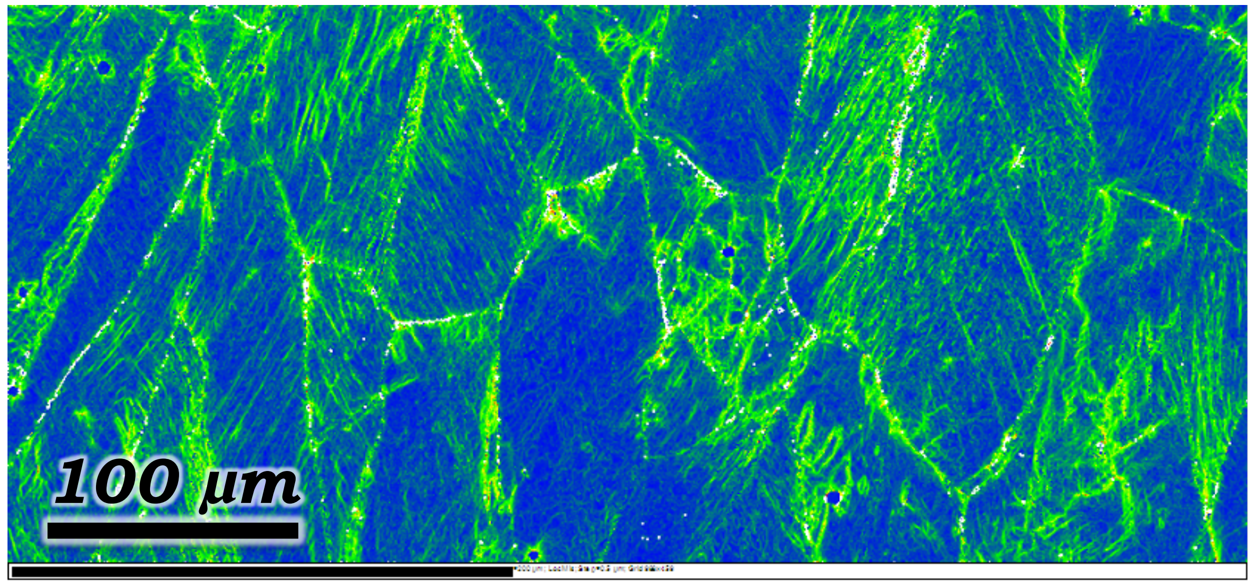
Cold rolled alloy 690SA has a lower degree of IGSCC engagement and growth, mainly large pockets of IG growth along the crack front.

EBSD-Indicated Strain in Highly Cold Rolled TT and SA Materials

Strain is linearly proportional to misorientation parameter

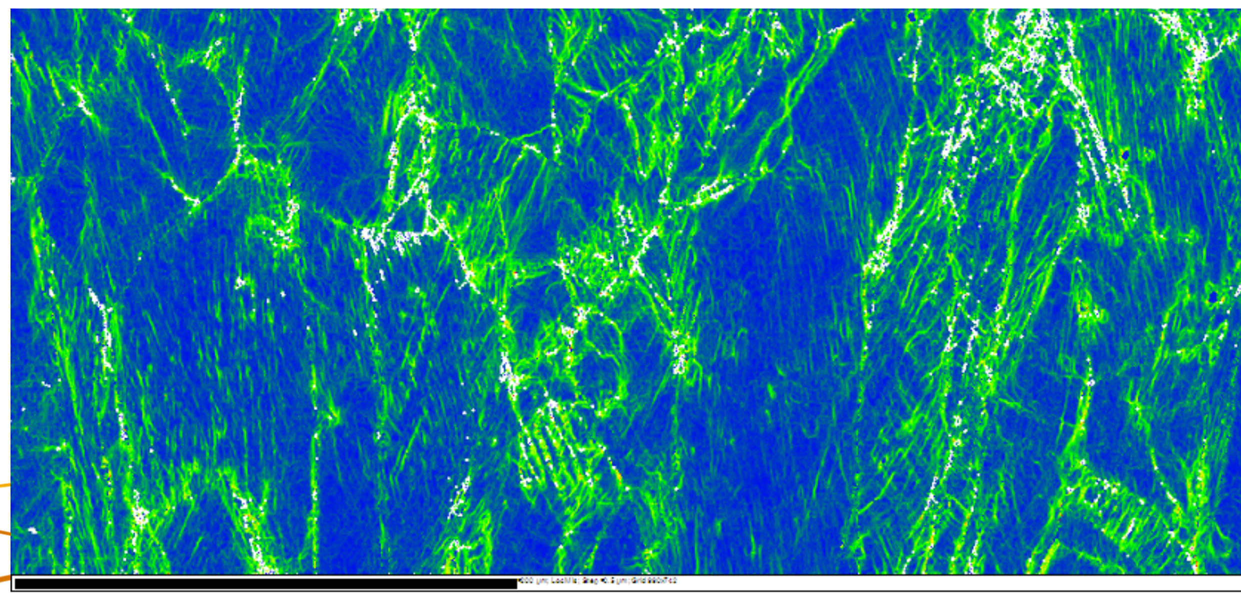
Misorientation represented by green intensity

31%CR Alloy 690TT 300 kg/mm² →

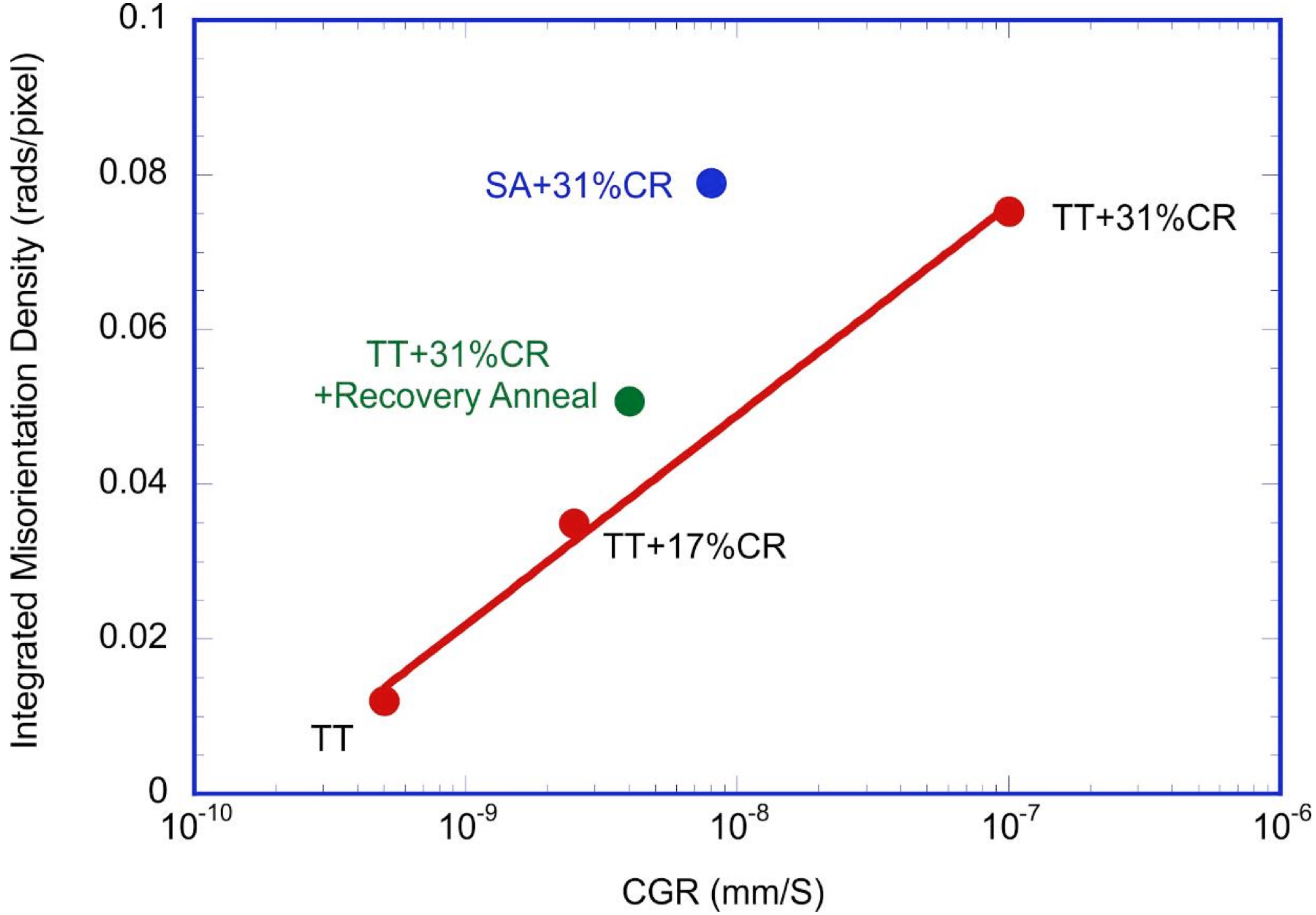


Similar level of indicated strain, perhaps more continuous GB distribution for TT.

31%CR Alloy 690SA 290 kg/mm² →



Influence of Cold Rolling on Strain Distributions for Alloy 690TT CRDM Tubing



A linear relationship is suggested between the integrated misorientation density and the measured SCC growth rates for the CR TT materials. The 31%CR SA does not follow this trend.

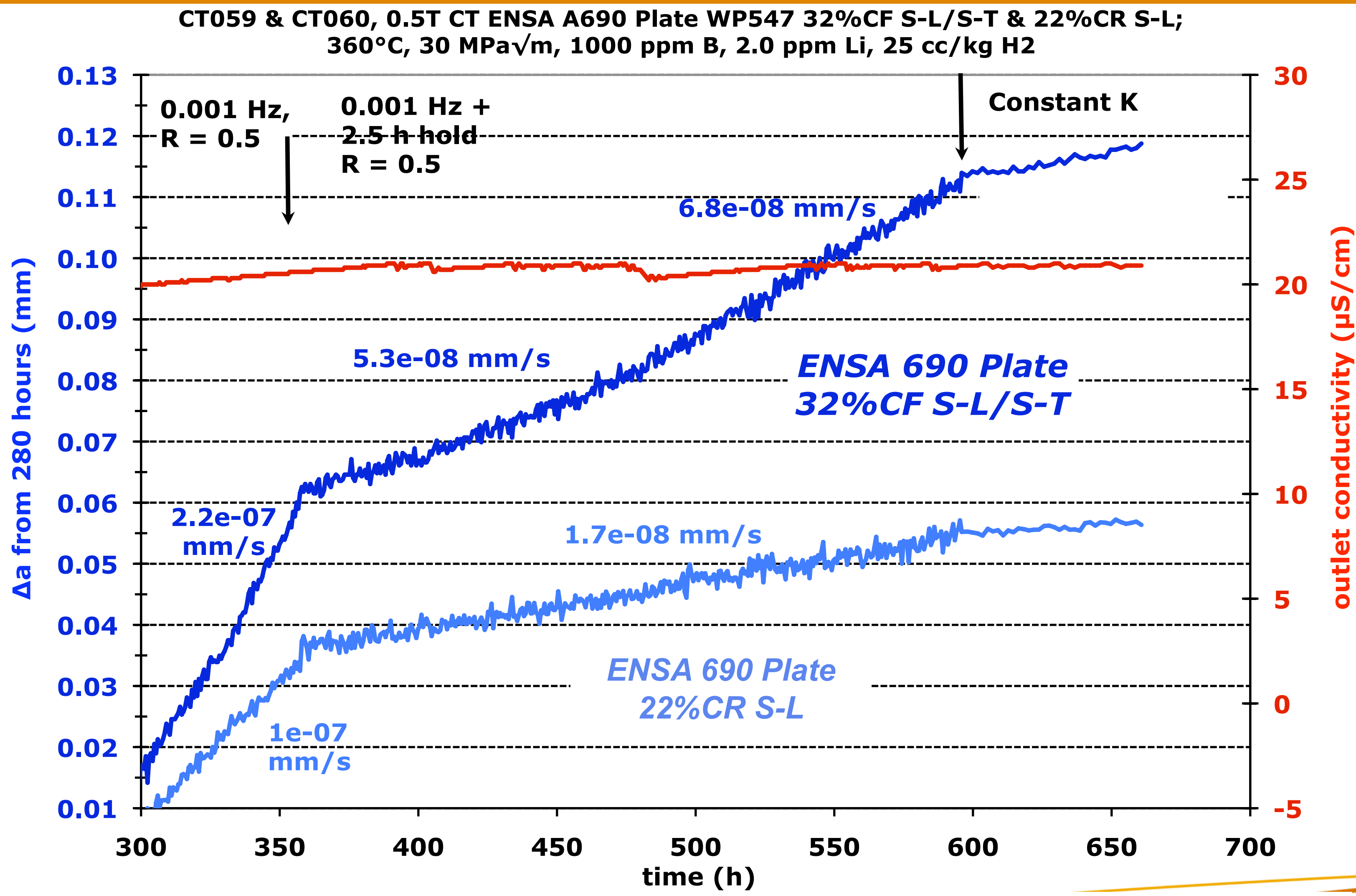
Microstructure and Cold Rolling Effects on Primary Water SCC of CRDM Alloy 690

- *Unidirectional cold rolling increases SCC crack growth rate and the degree of IG engagement for CRDM alloy 690TT (as-received) material in PWR primary water. CGRs increase ~10x for 17%CR S-L and ~200x for 31%CR S-L above the non-CR material.*
- *A 700°C/1 hour recovery heat treatment strongly reduces IGSCC of 31%CR alloy 690TT. This may result from a relaxation/ reorganization in the dislocation structure; treatment does not alter basic microstructure or permanent GB damage.*
- *Initial solution annealing to remove GB carbides produces a much lower susceptibility to IGSCC in highly cold-rolled materials. The difference in response between SA and TT is small at lower cold work levels, but constant K CGRs of highly CR alloy 690SA are consistently ~10X lower than alloy 690TT.*
- *Further research is underway to continue to study mechanisms controlling IGSCC susceptibility in cold worked alloy 690. Additional materials and conditions are being evaluated to isolate the influence of grain boundary carbide distribution and Cr composition both in this CRDM heat (RE243) and a plate heat.*

PNNL Alloy 690 Summary + Near Term Plans

- ***Microstructure and Cold Work Effects on SCC of Alloy 690 CRDM Tubing***
  - *Single heat of alloy 690 CRDM tubing: heat RE243*
  - *Cold rolling for TT vs SA: 0%, 17% (S-L), 30% (T-L), 31% (S-L)*
  - *Recovery anneal: 31%CR alloy 690TT + 700°C/1h/AC (S-L)*
  - ***Desensitized (900°C/1h) to keep GB carbides but remove most Cr depletion followed by 31%CR (S-L)***
- ***Microstructure and Cold Work Effects on SCC of Alloy 690 Plate Materials***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - ***Ongoing for 22%CR (S-L) and 30%CF (S-L/S-T) ENSA heat.***
  - ***SA versus TT for GE B25K heat followed by 20%CR.***

Early SCC Growth Rate Response for Cold Forged and Cold Rolled ENSA Plate



Initial transitioning completed and first constant K attempt is ongoing.
Higher growth rates during cycle + hold for 32%CF than 22%CR plate.

PNNL Alloy 690 Near Term Plans
GB Microstructure/Chemistry Effects

Material	TM Treatment – Material Condition	Purpose	Estimated Test Date
GE Plate B25K	Solution anneal (1100C/1h/WQ) + 20% 1D CR/CF	Remove matrix and grain boundary carbides (may alter if Cr segregated during WQ), evaluate GB carbide effects	2011-12
GE Plate B25K	Solution anneal + TT (720C/10h/AC) + 20% 1D CR/CF	Produce semi-continuous distribution of GB carbides, evaluate GB carbide effects	2011-12
Valinox Alloy 690 CRDM RE243	AR-TT + Desensitized (900C/0.5h/WQ) + 31% 1D CR/CF	Keep GB carbides, remove most Cr depletion, evaluate GB Cr concentration effects	2012
Valinox Alloy 690 CRDM RE243	Carbide modification (SA+900C and WQ) + 31% 1D CR/CF	Alter GB carbides and remove Cr depletion, evaluate GB carbide effects	2012

PNNL Alloy 690 Near Term Plans
Cold Work and Heat-to-Heat Effects

Material	TM Treatment – Material Condition	Purpose	Estimated Test Date
Valinox Alloy 690 CRDM RE243	As-received TT + ~30% CF	Evaluate CW effects and method of deformation	2011-12
Valinox Alloy 690 CRDM RE243	As-received TT + ~30% Tensile Strain	Evaluate CW effects and method of deformation	2011-12
Alternative Alloy 690 CRDM #4	As-received TT + 20-30% CF	Evaluate CW and heat-to-heat effects	2012
Alternative Alloy 690 CRDM #4	As-received TT + ~20% Tensile Strain	Evaluate CW and heat-to-heat effects	2012
Valinox Alloy 690 CRDM RE243	As-received TT + ~20% Tensile Strain	Evaluate CW effects and critical deformation level	2012
Alternative Alloy 690 CRDM Material #5	As-received TT + ~20% CW	Evaluate heat-to-heat and initial microstructure	2012
Alternative Alloy 690 CRDM Material #6	As-received TT + ~20% CW	Evaluate heat-to-heat and initial microstructure	2012