Microstructural Characterizations of Alloy 690: Key Examples

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PNNL Characterization Actvities: Examples for Alloy 690 Materials

Microstructural Characterization

- *Essential for material assessment and comparisons including heat-to-heat, processing and heat treatment effects.*
- *Important to assess general microstructure (grain size/shape, banding), precipitate microstructures (size/distribution IG and TG), local microchemistry (grain boundary depletion/segregation), matrix hardness and strain distributions.*
- *Open question how detailed characterizations should be on most materials, depends on specific issues being examined.*

Characterization Methods

- *Optical metallography, SEM and EBSD for general microstructure*
- *SEM and TEM for precipitate microstructure*
- *TEM for grain boundary microchemistry and phase identification*
- *EBSD for strain distributions*
- *Optical, SEM and TEM of SCC cracks and crack tips*

PNNL Characterization Actvities: Examples for Alloy 690 Materials

- ***Initial Alloy 690 Microstructures***
  - *As-received thermally treated (TT), solution annealed (SA) or desensitized alloy 690 CRDM tubing*
  - *As-received alloy 690 mill annealed (MA) plate heats*
- ***Cold Work Effects on Alloy 690 Microstructures***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - *17-31%CR CRDM alloy 690TT and alloy 690SA, influence of recovery anneal after 31%CR alloy 690TT (heat RE243)*
- ***SCC Crack and Crack-Tip Exams on Alloy 690***
  - *SCC morphology and crack path interactions with CR damage*

Microstructure Characterizations for CRDM Alloy 690 Heat RE243

Alloy 690 CRDM Heat RE243: As-received, thermally treated (TT) condition

Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Equiaxed grains, avg. size ~100 μm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Content	TEM-EDS	Cr minimum ~21-24 wt%, width 300-400 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 μm)
Hardness	Vickers	175 kg/mm^2 (CT crack plane)
Damage μS	SEM/TEM	None, low dislocation density
Strain & GB Distribution	EBSD	Low strain levels, random GBs >75%

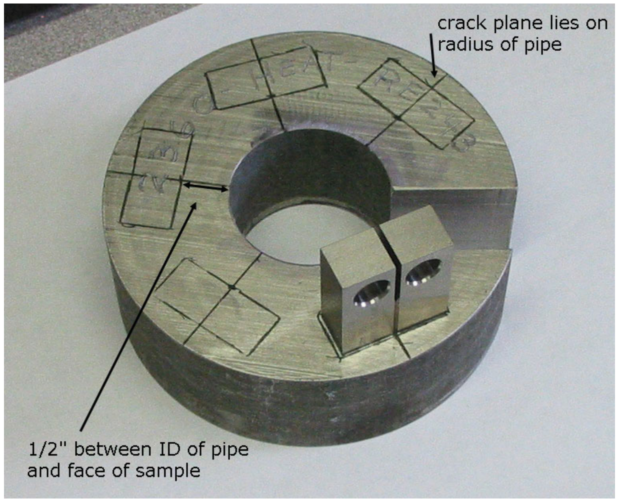
Alloy 690 CRDM Heat RE243: Solution-annealed (SA) condition

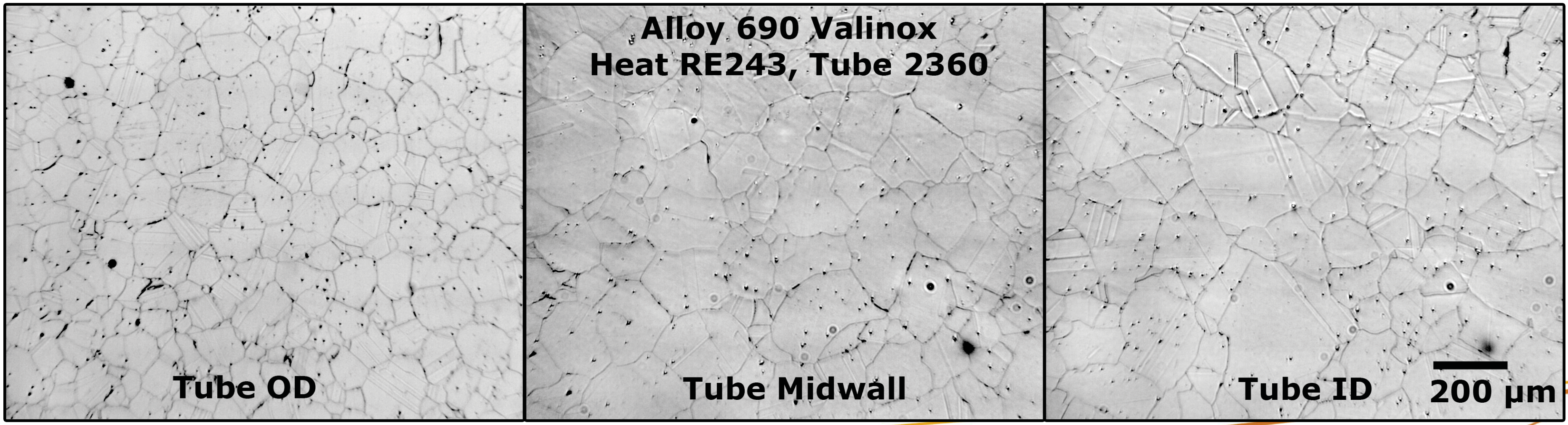
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Equiaxed grains, avg. size ~120 μm
IG Precipitates	OM/SEM/TEM	Few, isolated $M_{23}C_6$, occasional TiN (~500 nm)
GB Cr Content	TEM-EDS	30 wt%, no depletion or enrichment
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 μm)
Hardness	Vickers	165 kg/mm^2 (CT crack plane)
Damage μS	SEM/TEM	None, low dislocation density
Strain Dist.	EBSD	Very low strain levels

Isolating effect of grain boundary Cr carbides.

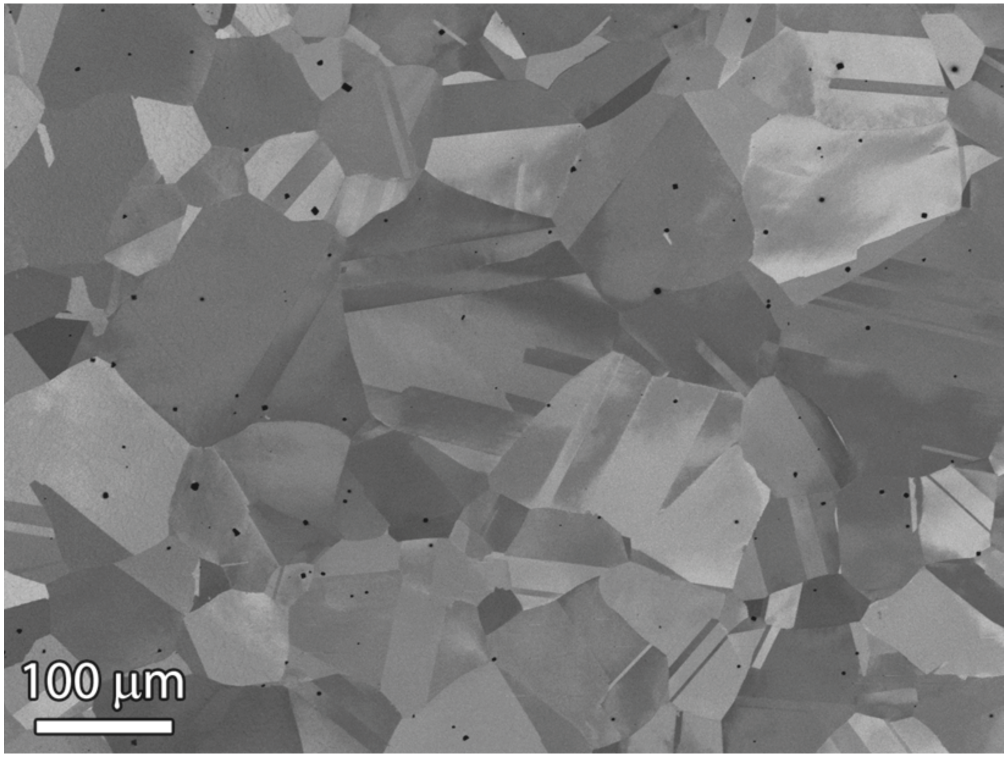
As-Received, Alloy 690TT CRDM Tubing

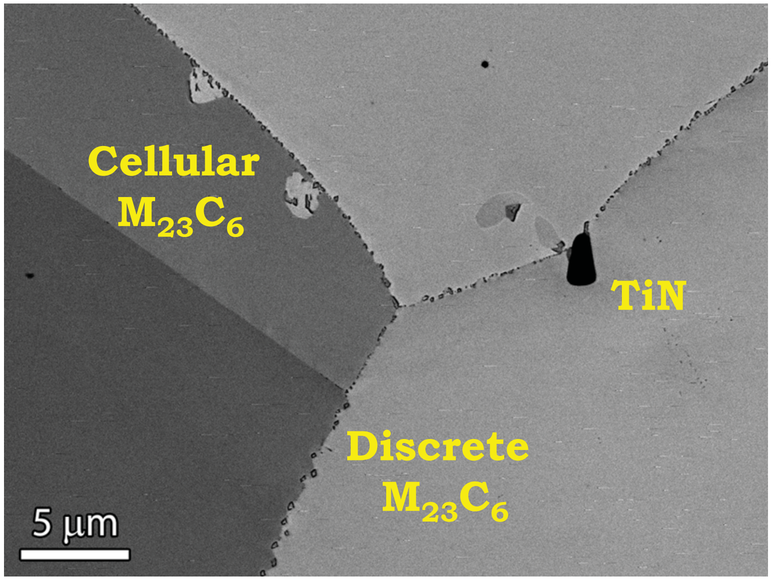
- *Extruded tubing provided by Valinox in a thermally treated condition (720°C/ 10.5 hr)*
- *Equiaxed grain dimensions (70-120 µm) at midwall and ID, smaller very near tube OD*
- *High density of nearly continuous, grain boundary carbides*
- *Low density of matrix TiN particles*
- *No banded structures*

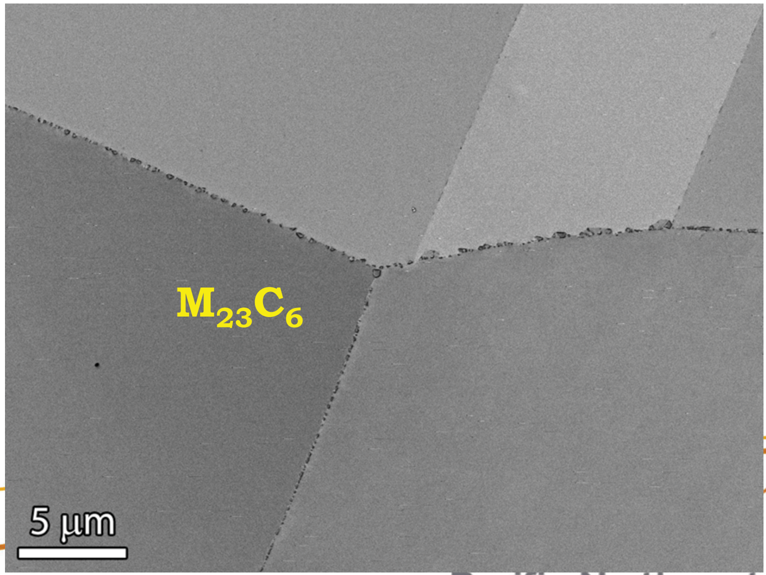




Grain Boundary Microstructures in As Received, Thermally Treated (TT) Alloy 690

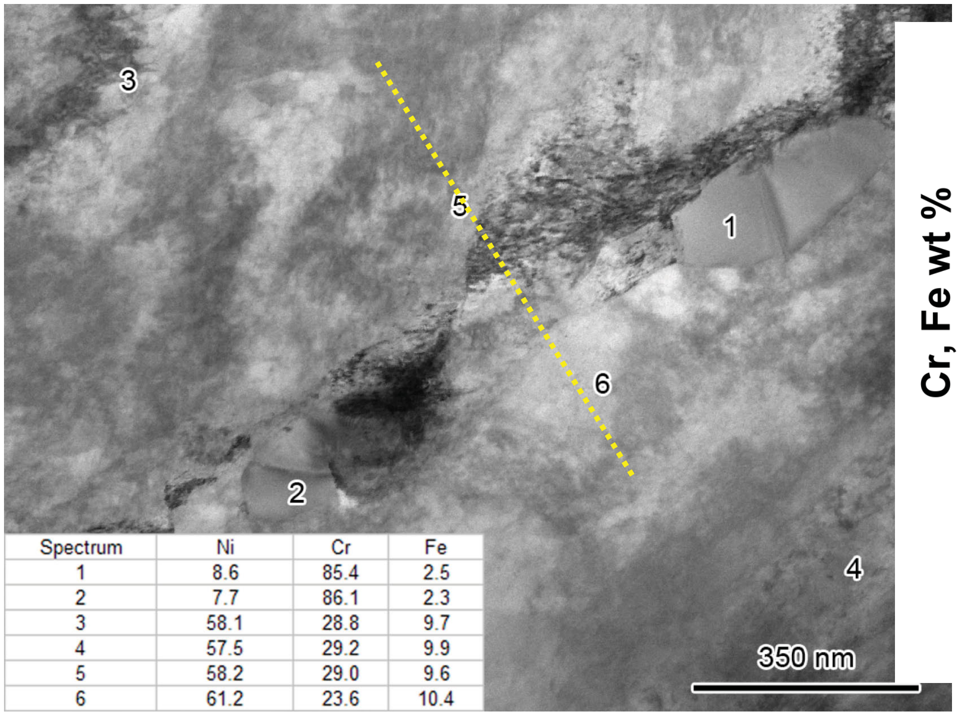




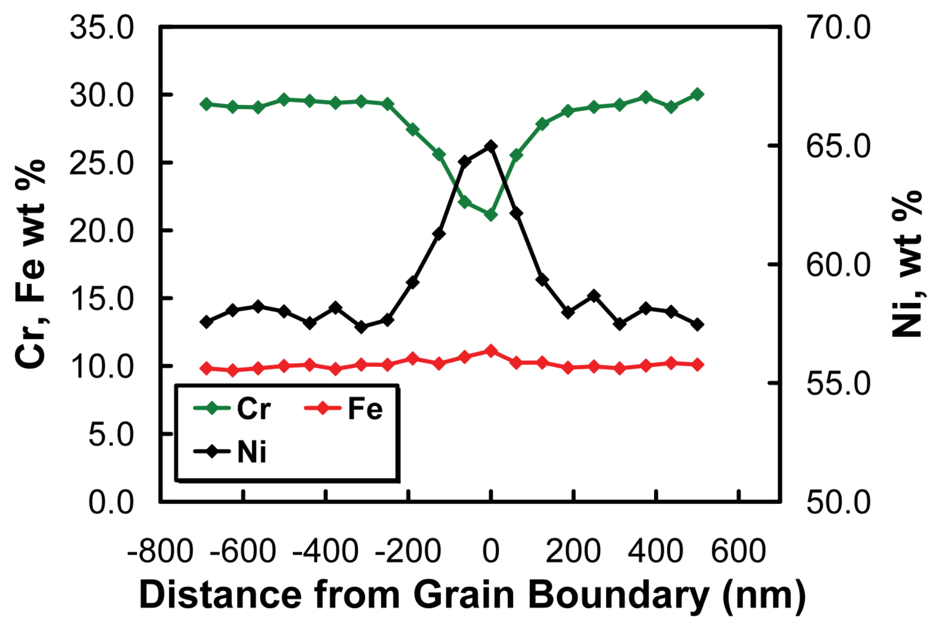


SEM backscatter images show general microstructure and grain boundary carbides in alloy 690TT CRDM tubing heat RE243.

Grain Boundary Cr Depletion in As-Received Alloy 690TT CRDM Tubing Heat RE243

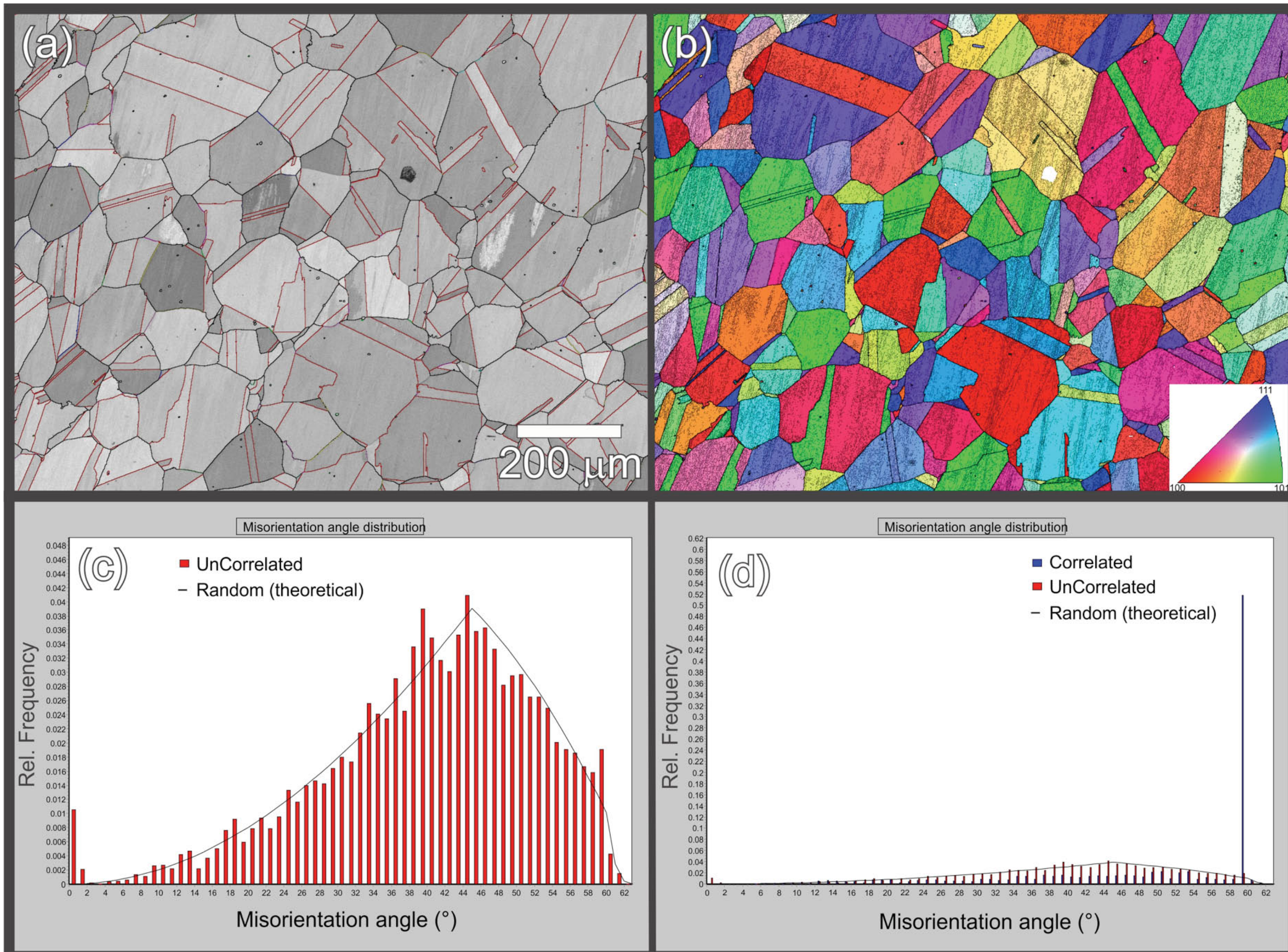


Spectrum	Ni	Cr	Fe
1	8.6	85.4	2.5
2	7.7	86.1	2.3
3	58.1	28.8	9.7
4	57.5	29.2	9.9
5	58.2	29.0	9.6
6	61.2	23.6	10.4



Semi-continuous GB carbides for the as-received alloy 690TT material with significant Cr depletion observed associated with both discrete carbides and cellular $M_{26}C_6$ precipitates.

As-Received, Alloy 690TT CRDM Tubing



EBSD shows a high density of twin boundaries, but most grain boundaries (>75%) are random, high-energy boundaries.

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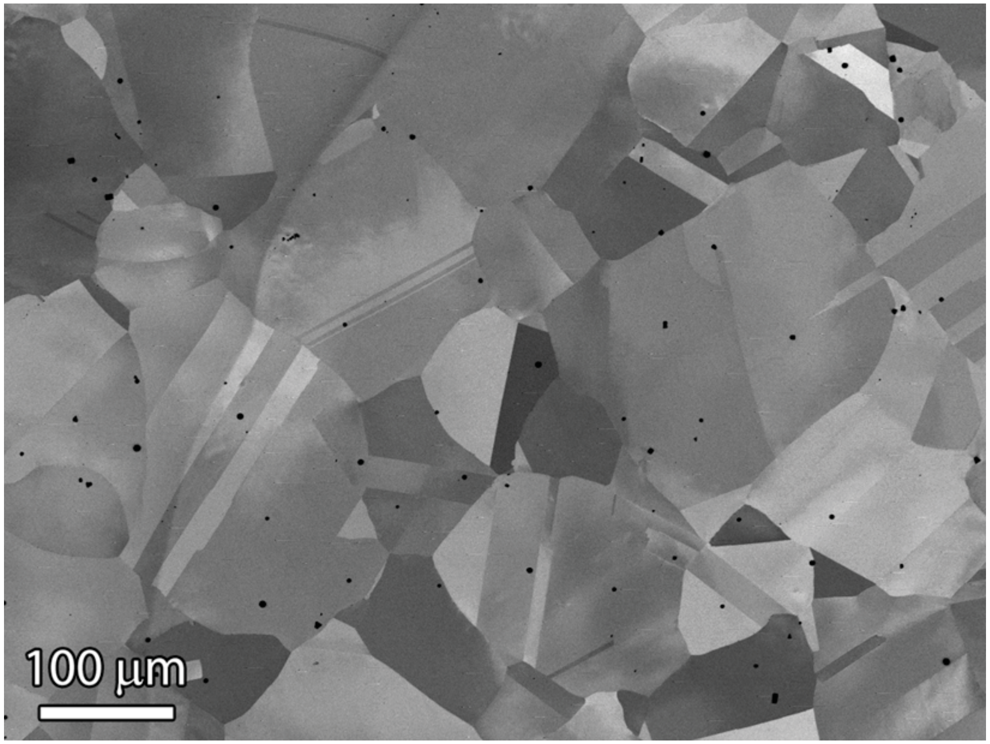
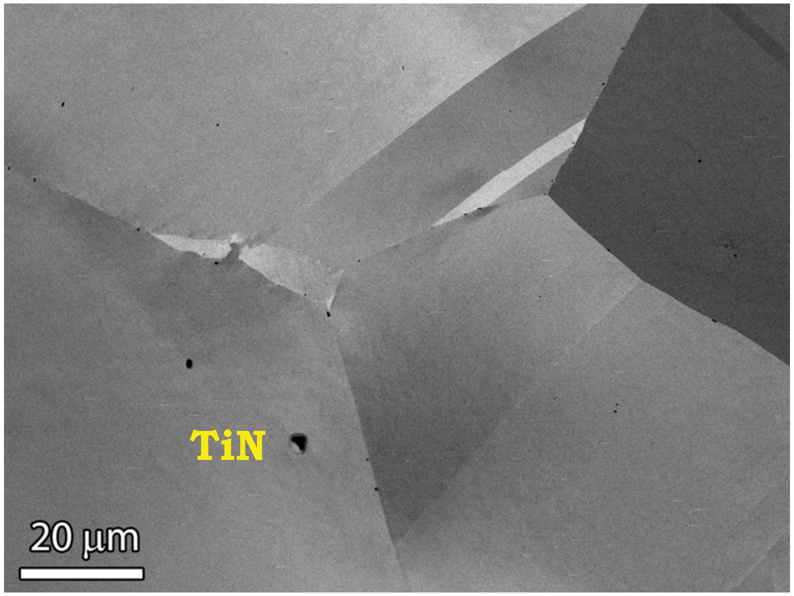
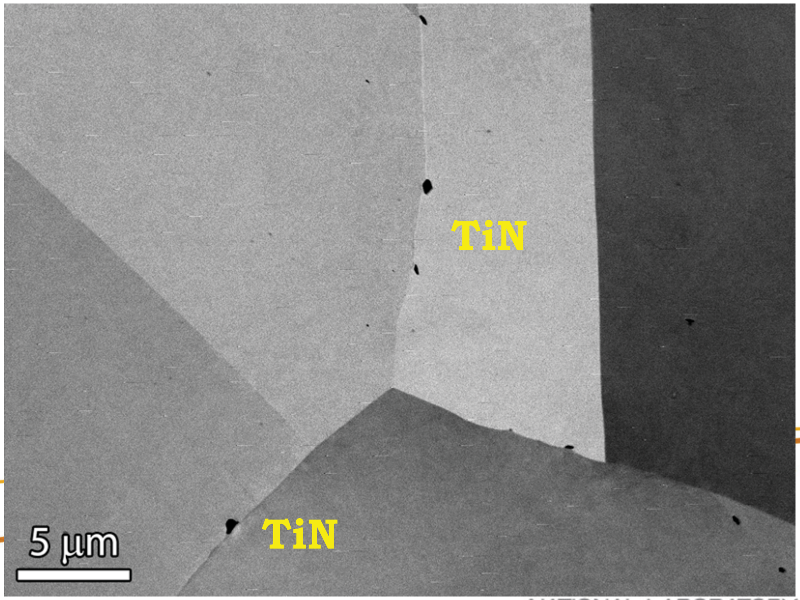
Grain Boundary Microstructures in Solution Annealed (SA) Alloy 690



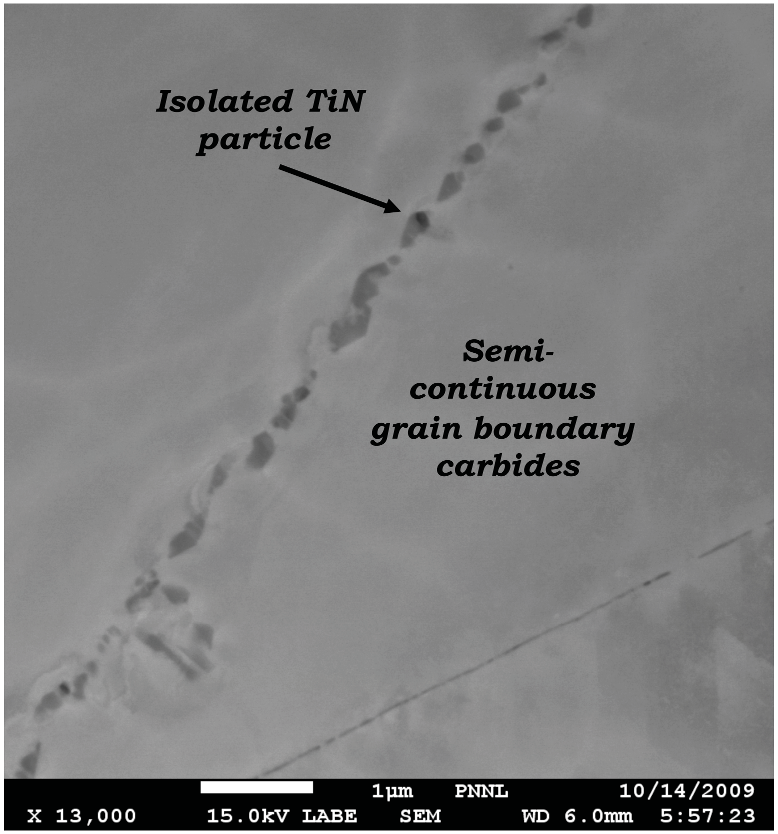




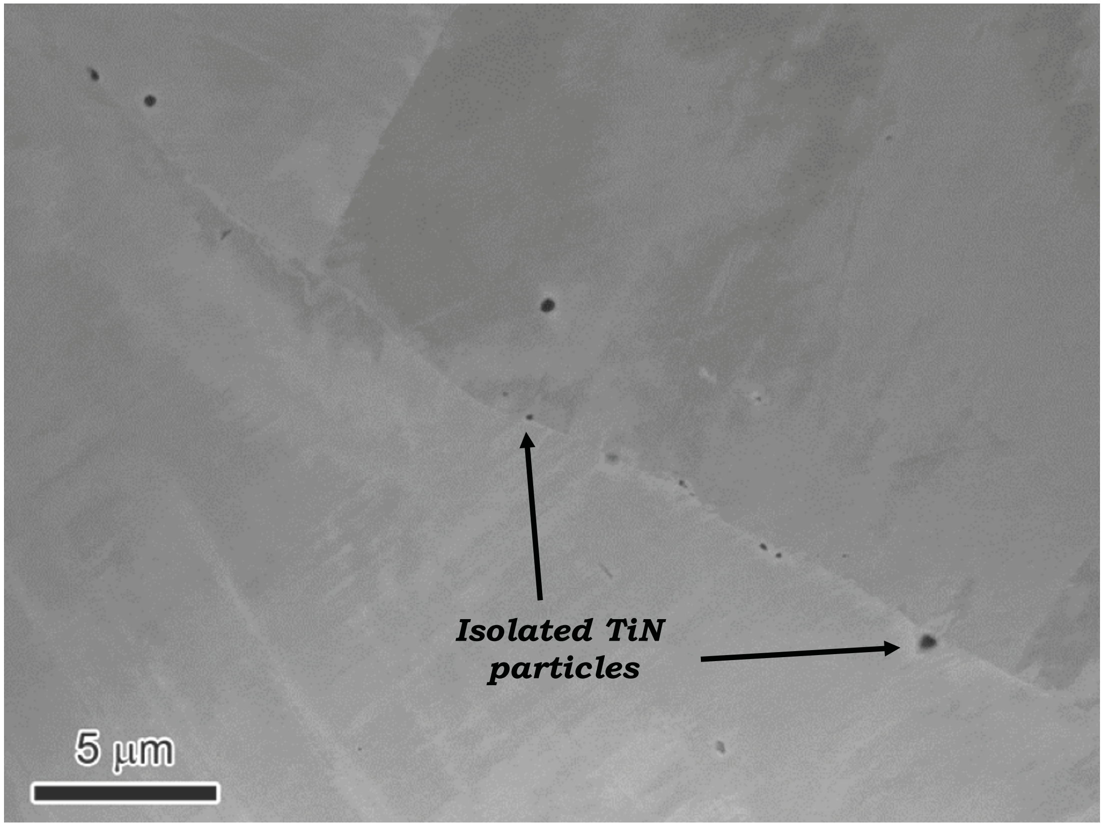
Solution anneal at 1100°C and water quench removed nearly all grain boundary carbides in CRDM heat RE243, isolated TiN particles remain. Slight increase in average grain size.

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

Alloy 690TT CRDM



Alloy 690TT + SA



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

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Microstructure Characterizations for CRDM Alloy 690 Heat RE243

Alloy 690 CRDM Heat RE243: As-received, thermally treated (TT) condition

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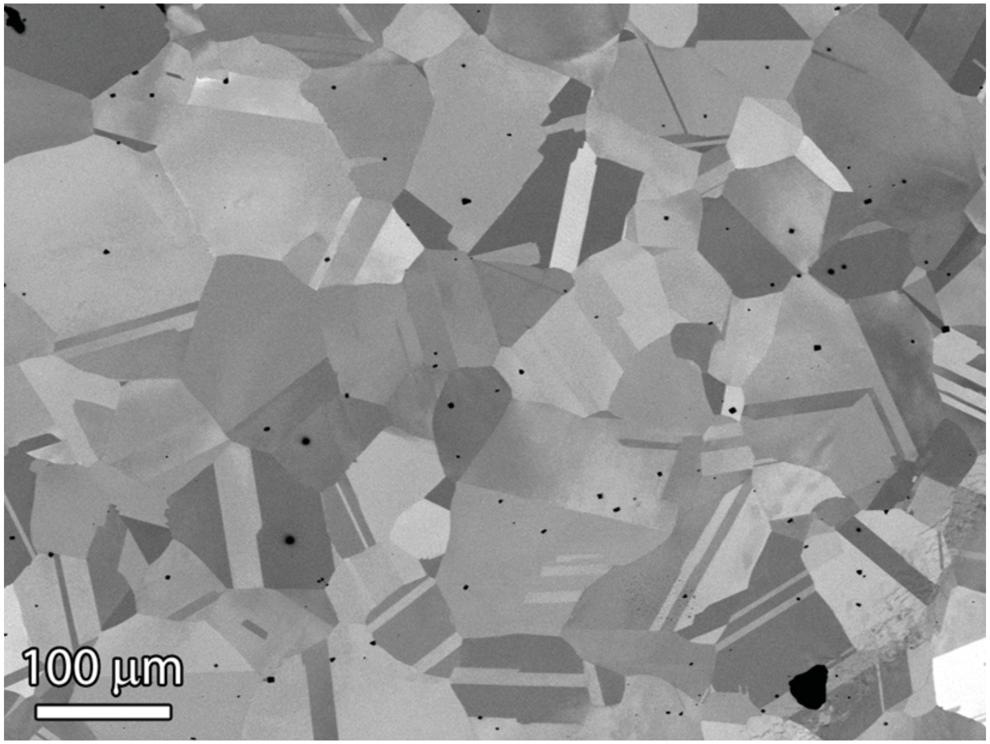
Grain Shape/Size	OM	Equiaxed grains, avg. size ~100 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Content	TEM-EDS	Cr minimum ~21-24 wt%, width 300-400 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	175 kg/mm^2 (CT crack plane)
Damage µS	SEM/TEM	None, low dislocation density
Strain & GB Distribution	EBSD	Low strain levels, random GBs >75%

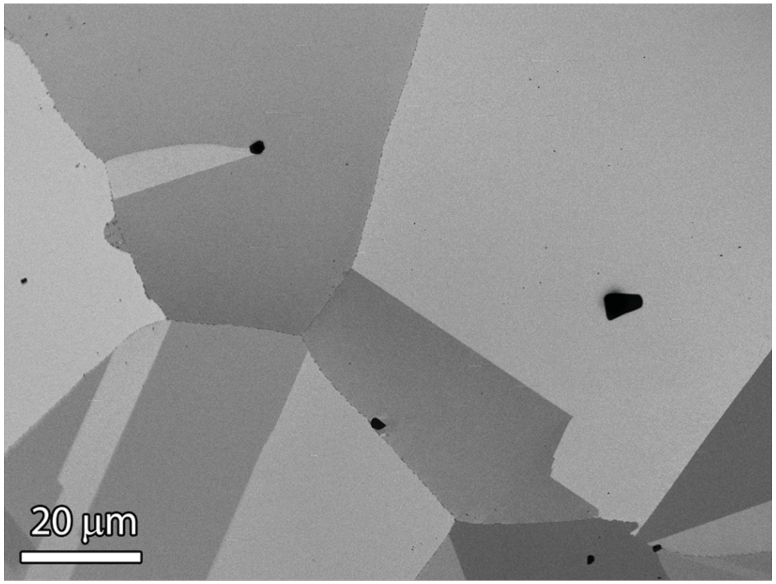
Alloy 690 CRDM Heat RE243: TT + desensitized condition

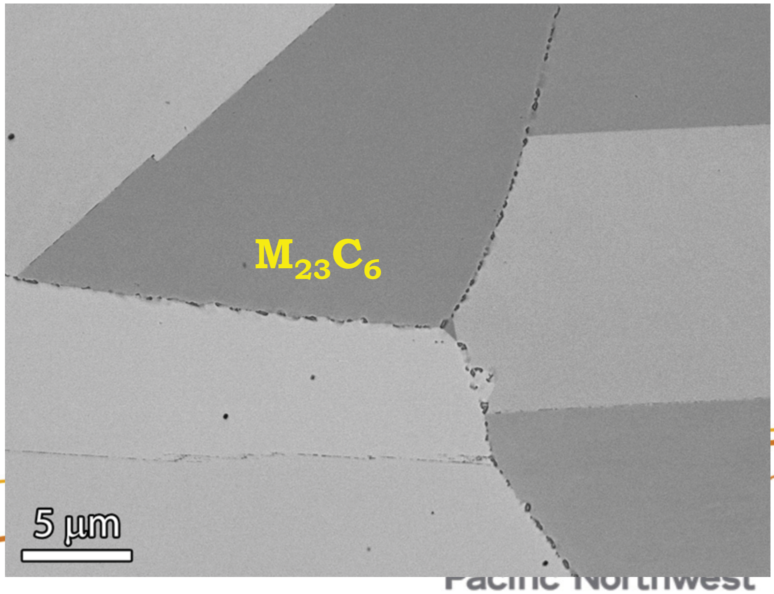
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Equiaxed grains, avg. size ~100 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-350 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Content	TEM-EDS	Cr minimum ~28 wt%, width ~1000 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	
Damage µS	SEM/TEM	None, low dislocation density
Strain Dist.	EBSD	Very low strain levels

Isolating effect of grain boundary Cr depletion.

Grain Boundary Microstructures in Desensitized Alloy 690

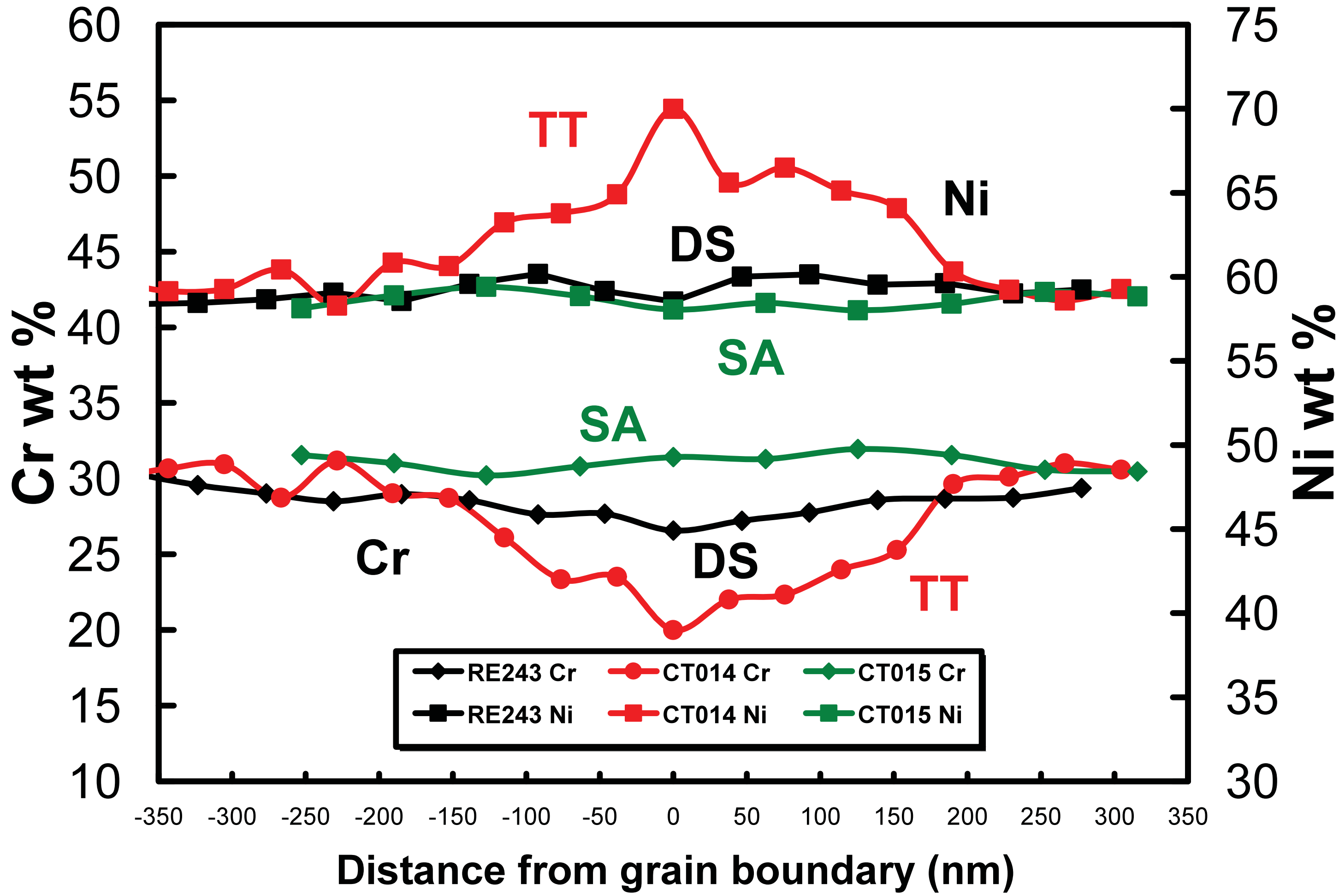






Desensitization heat treatment at 900ºC/ 1h and water quench keeps semi-continuous distribution of grain boundary carbides in CRDM heat RE243. Slight increase in carbide size and decrease in density.

Grain Boundary Cr Concentrations Alloy 690 CRDM Heat RE243



Solution anneal removes GB carbides and Cr depletion, desensitization keep GB carbides but removes most Cr depletion.

PNNL Characterization Actvities: Examples for Alloy 690 Materials

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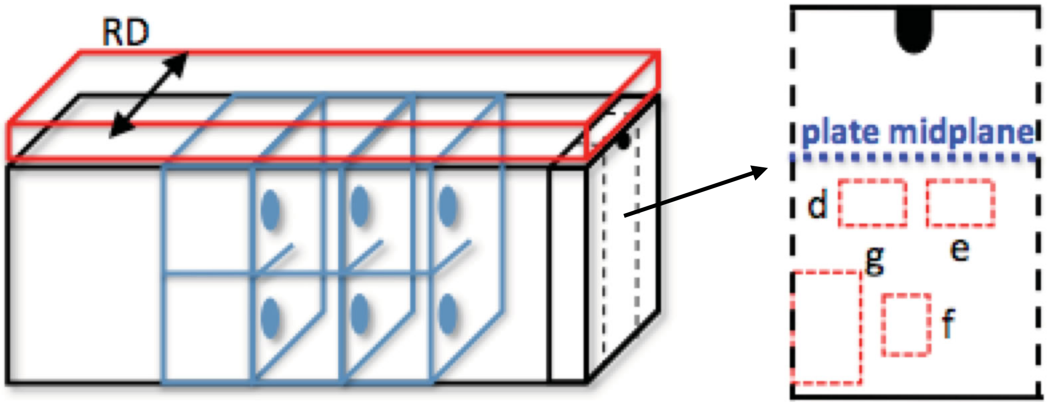
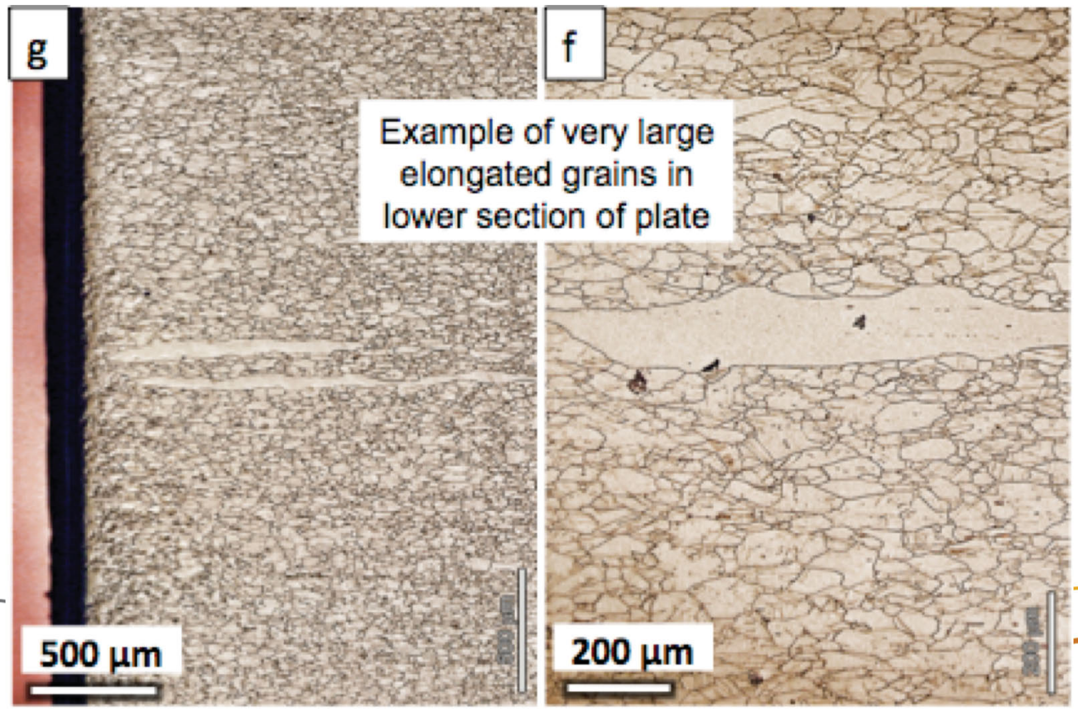
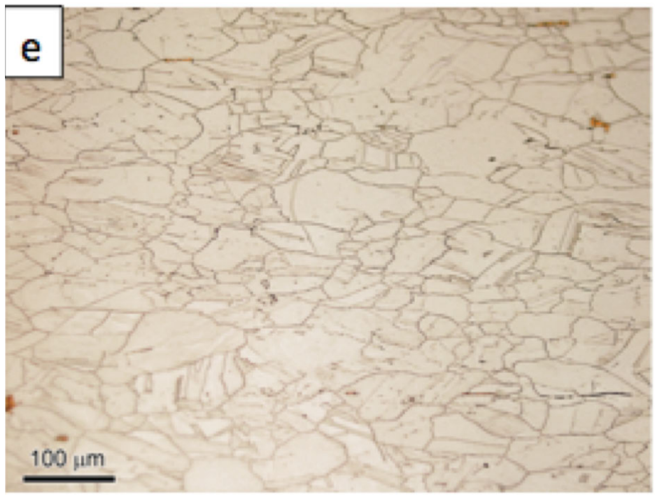
- ***SCC Crack and Crack-Tip Exams on Alloy 690***
  - *SCC morphology and crack path interactions with CR damage*

Example Microstructures: ANL 26%CR Plate at Crack Plane

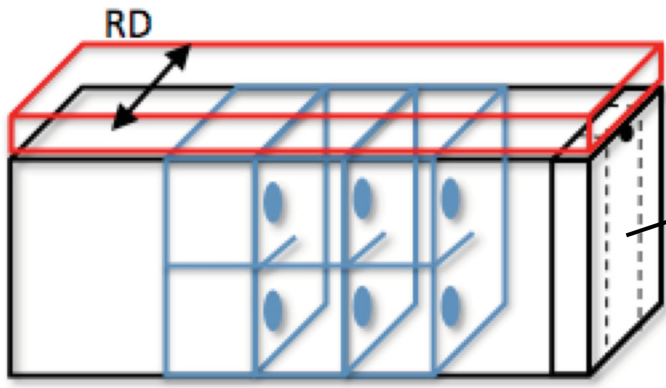


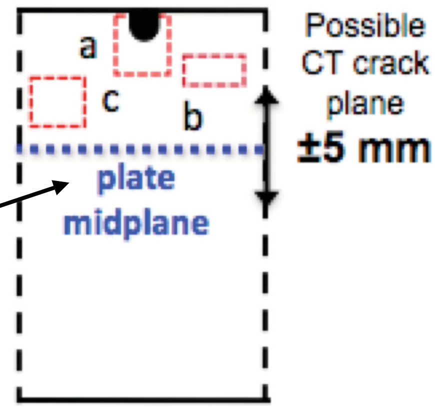
Evidence for compositional banding found in lower region of plate. Consistent microstructure and no banding in region corresponding to PNNL specimen crack growth plane





Example Microstructures: ANL 26%CR Plate at Upper Region of Plate

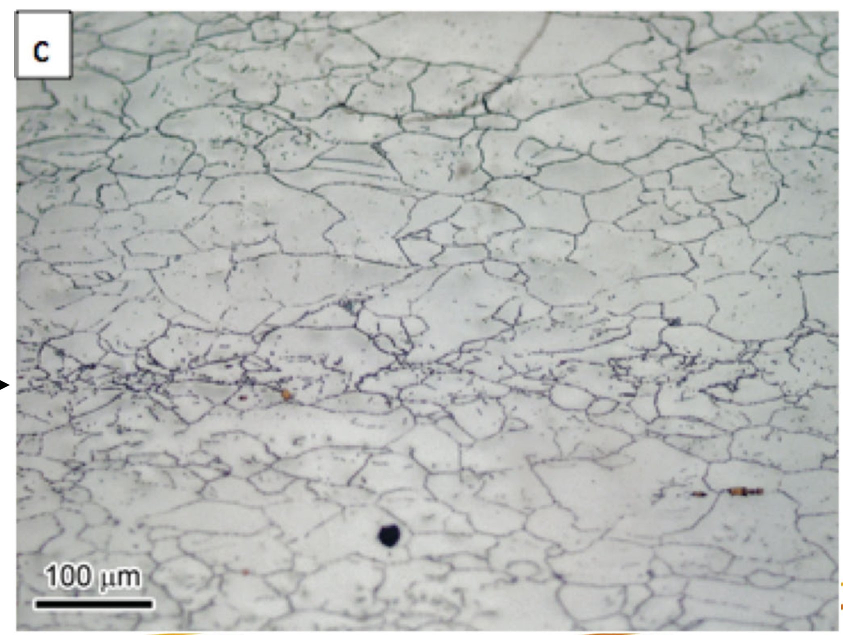




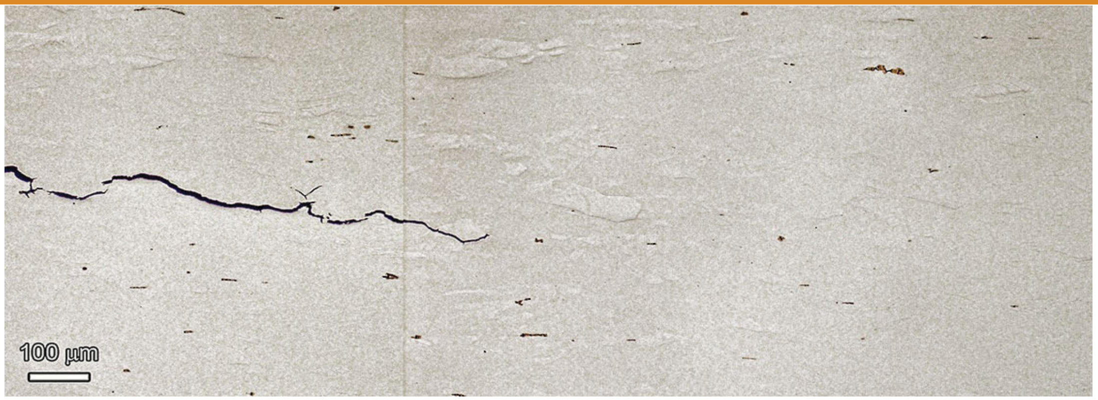
Evidence for compositional banding found in upper region of plate.

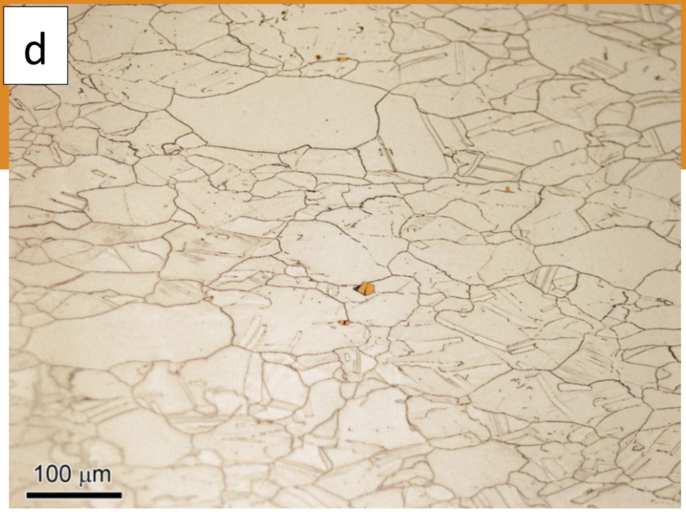
Band of larger grains

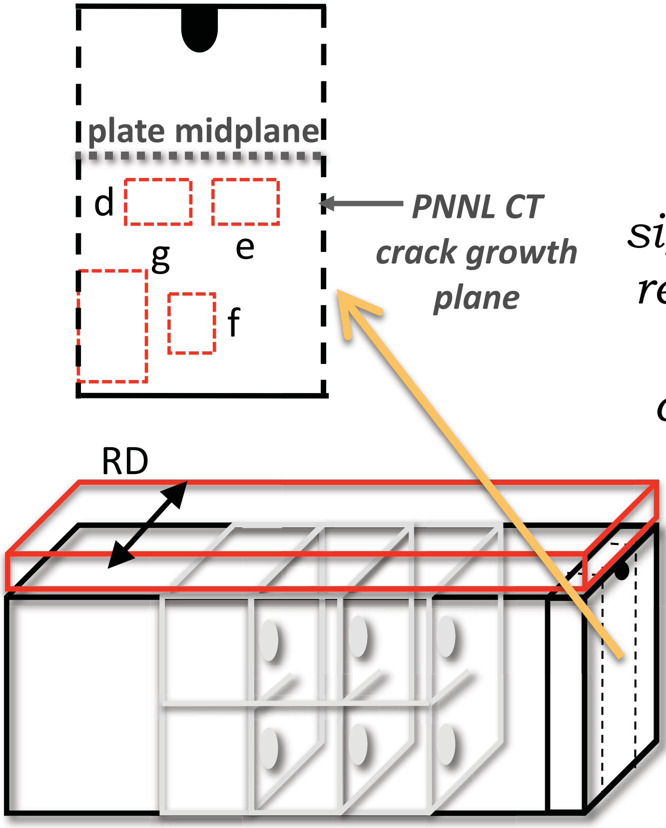
finer grains, high density of carbides



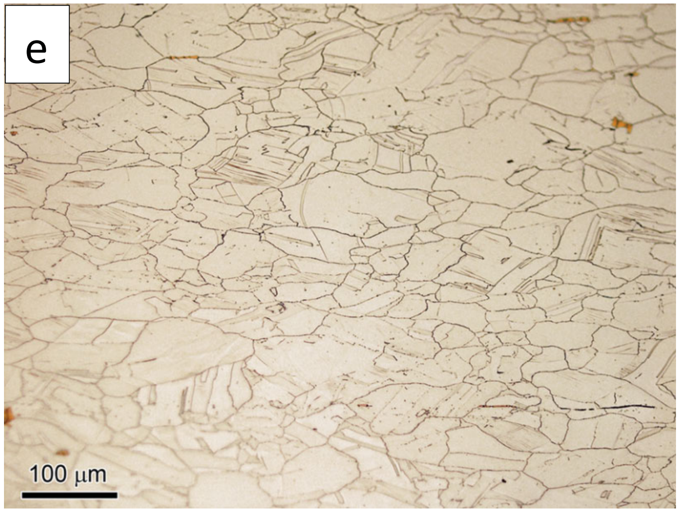
Example Microstructures: ANL 26%CR Plate at Crack Plane



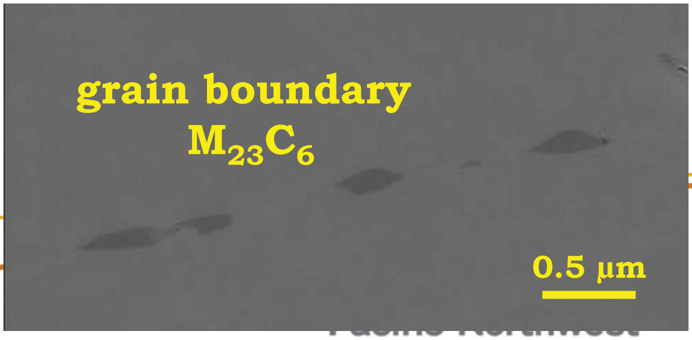




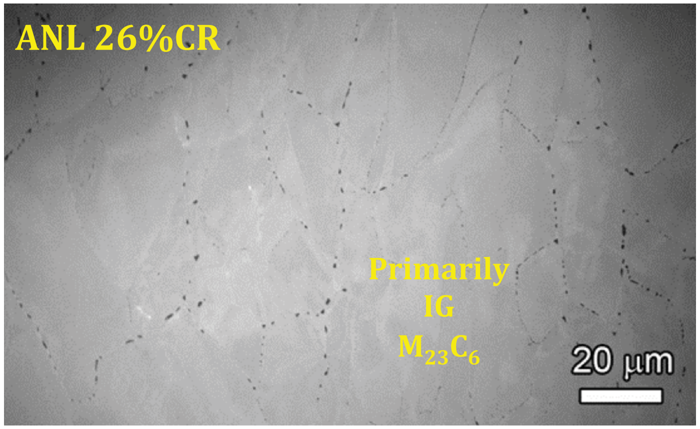
Consistent microstructure, no indication of significant banding in region corresponding to PNNL specimen crack growth plane

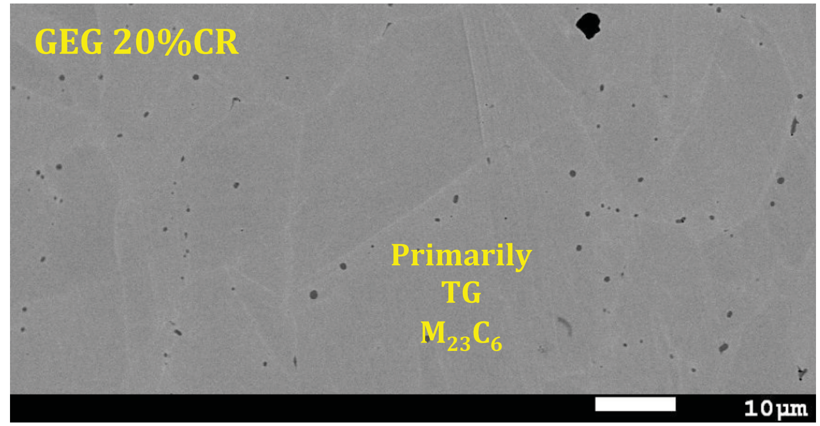


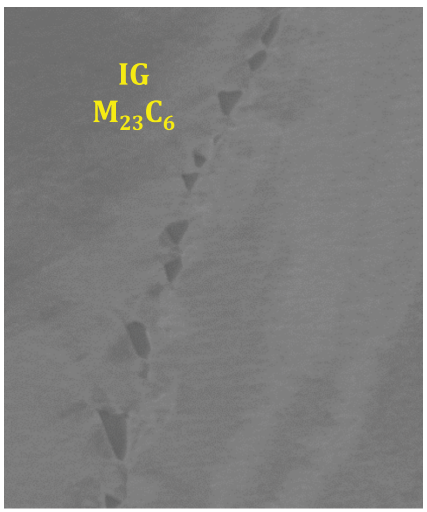
Moderate-to-high density of grain boundary carbides

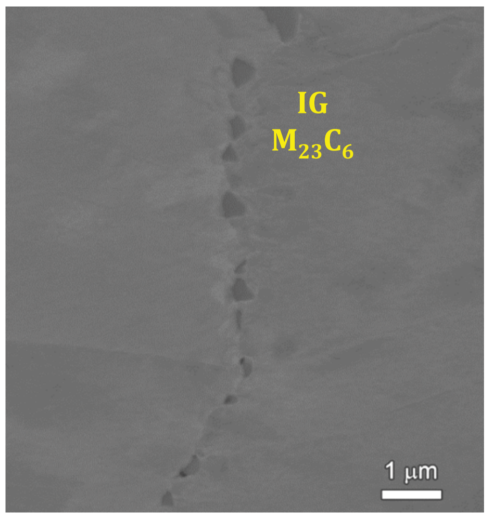


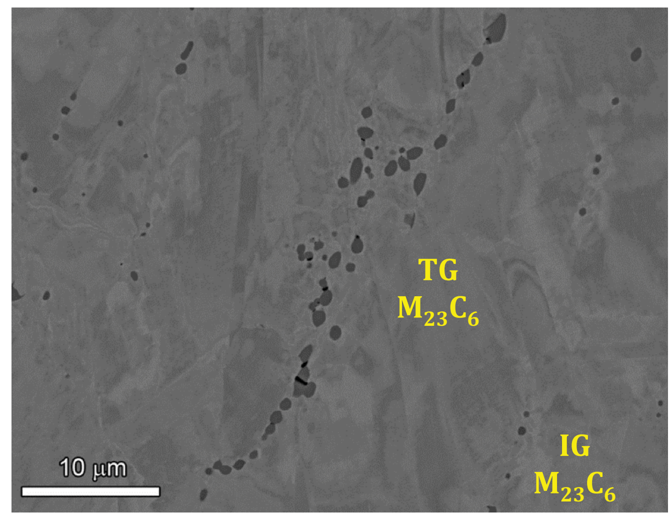
Precipitation Microstructures in ANL 26%CR and GEG 20%CR Alloy 690 Plates





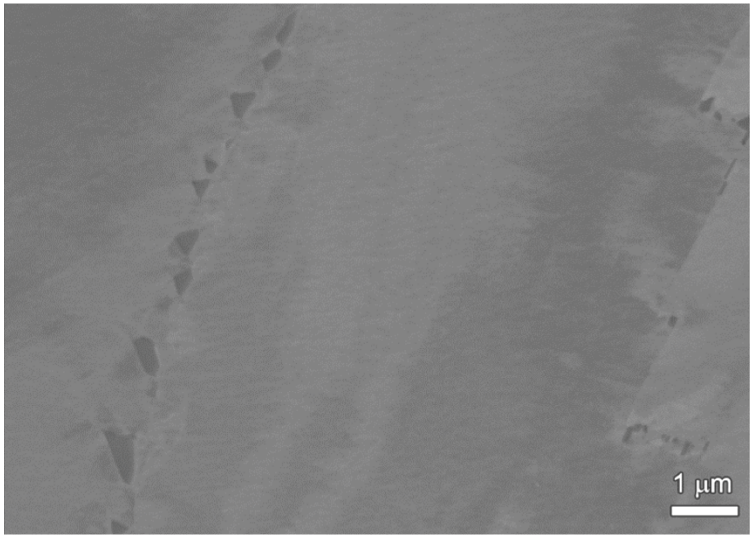




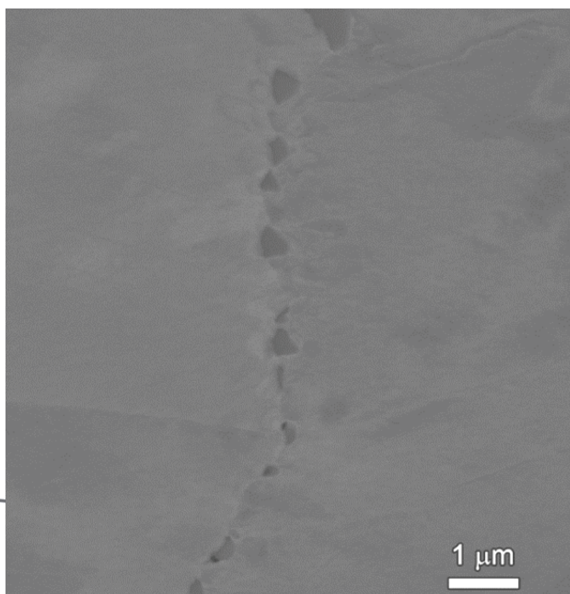


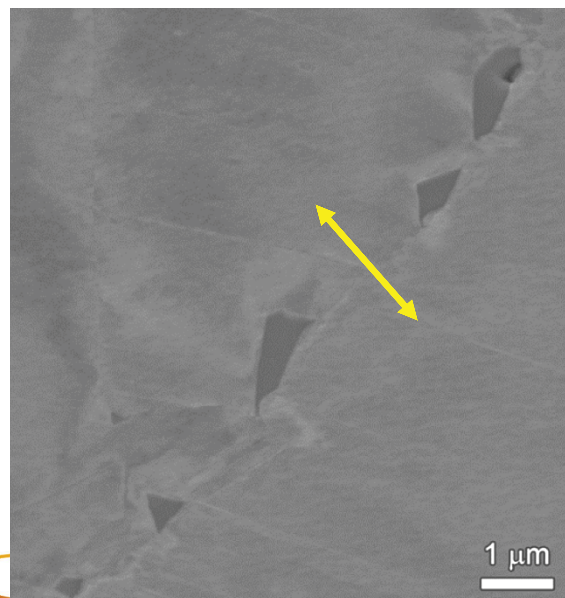
SEM examinations reveal that the ANL alloy 690 plate has a high density of Cr carbides at <u>nearly all</u> high-energy grain boundaries, while the GEG plate has a low density of carbides on <u>most</u> grain boundaries.

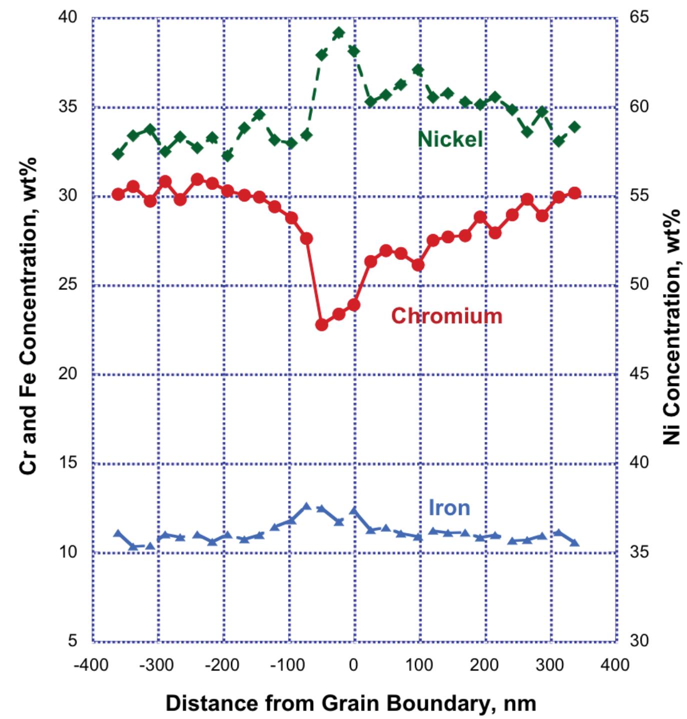
Grain Boundary Microstructure/Chemistry for ANL 26%CR Plate at Crack Plane



Most random grain boundaries have a high density of discrete $M_{23}C_6$ carbides and significant Cr depletion.







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- ***SCC Crack and Crack-Tip Exams on Alloy 690***
  - *SCC morphology and crack path interactions with CR damage*

Microstructure Characterizations for CR Alloy 690: ANL and GEG Plate Heats

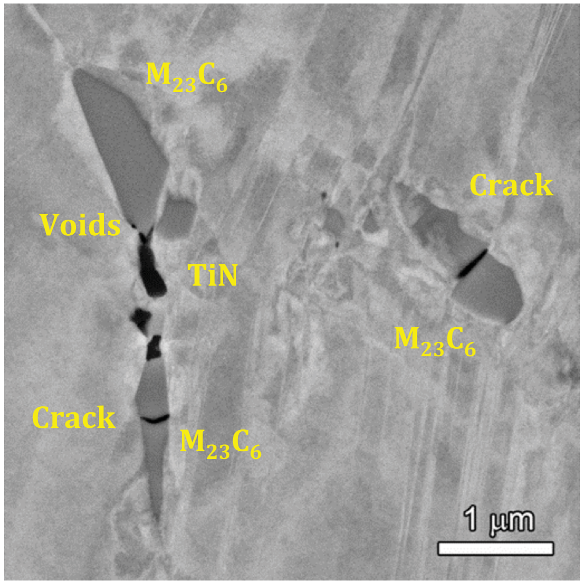
Alloy 690 Bar Heat NX3297HK12 (ANL): As-received, MA condition + 26%CR

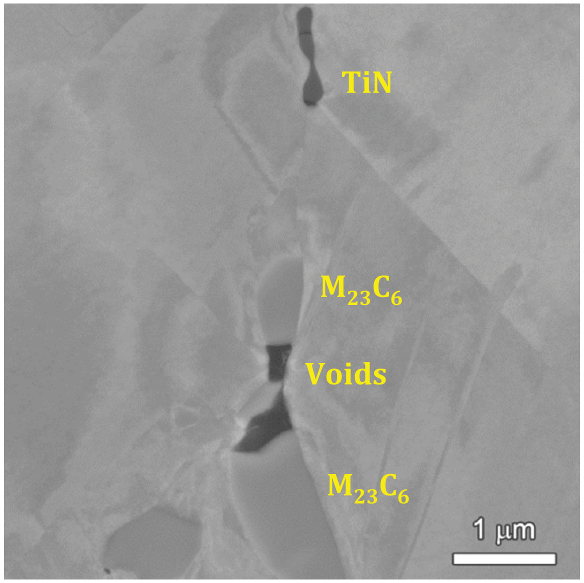
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Slightly elongated grains, avg. size ~80 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (0.1-1 µm) $M_{23}C_6$, occasional TiN (300-500 nm)
GB Cr Depletion	TEM-EDS	Cr minimum ~22 wt%, width 100-300 nm
TG Precipitates	OM/SEM	Many TiN (~1 µm), often as stringers
Banding	OM/SEM	Fine grains with higher density of second phases near top and bottom of plate
Hardness	Vickers	315 kg/mm^2 (PNNL CT crack plane)
Damage µS	SEM/TEM	High dislocation density, many GB cracked carbides and voids, many cracked matrix TiN
Strain Distribution	EBSD	High strain levels in matrix increasing at GBs, also local high strain at large matrix nitrides.

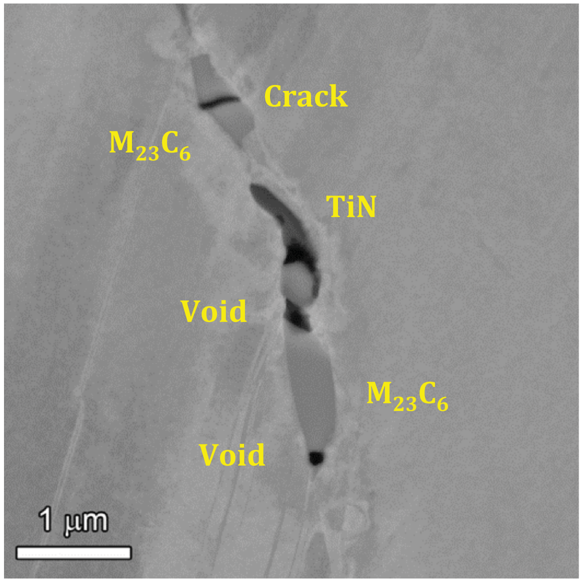
Alloy 690 Bar Heat B25K (GEG): As-received, MA condition + 20%CR

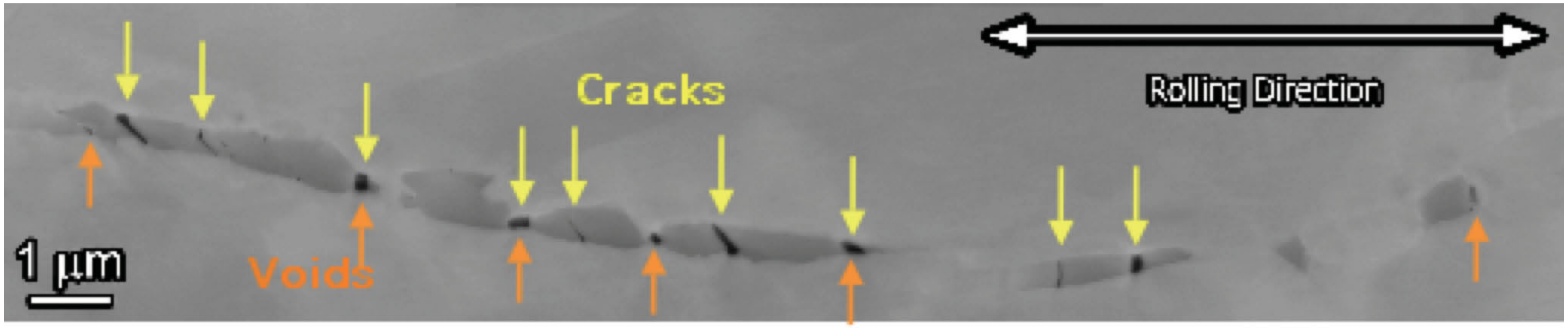
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Slightly elongated grains, avg. size ~70 µm
IG Precipitates	OM/SEM/TEM	Well-spaced, discrete (100-500 nm) $M_{23}C_6$, higher density in areas, few TiN (200 nm)
GB Cr Depletion	TEM-EDS	None for most GBs, certain GBs show areas with $M_{23}C_6$ and some depletion
TG Precipitates	OM/SEM	Many discrete (100-500 nm) $M_{23}C_6$, few random TiN or Ti carbonitrides (1-10 µm)
Banding	OM/SEM	TiN or Ti(C,N) perpendicular to cold rolling and to CT crack-growth plane
Hardness	Vickers	307 kg/mm^2 (CT crack plane)
Damage µS	SEM/TEM	High dislocation density, low density of GB voids, cracked GB $M_{23}C_6$, few cracked TiN
Strain Dist.	EBSD	Moderate strain in matrix increasing to GBs

Grain Boundary Damage Microstructures in Cold Rolled ANL 26%CR Alloy 690



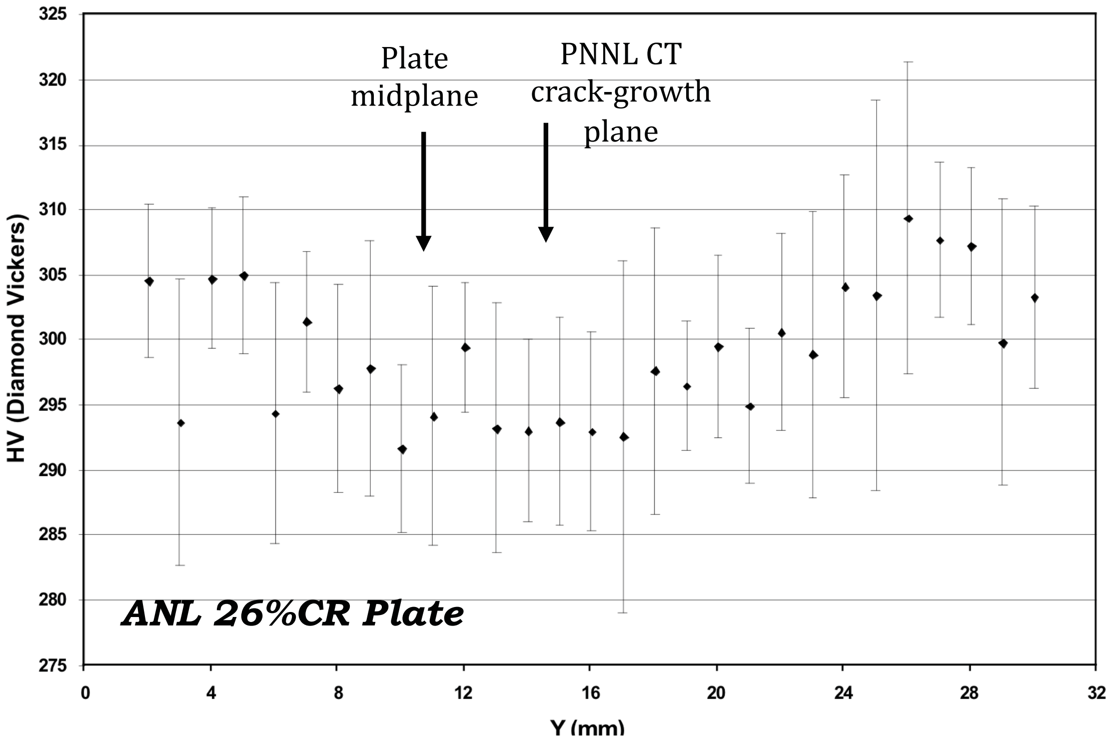


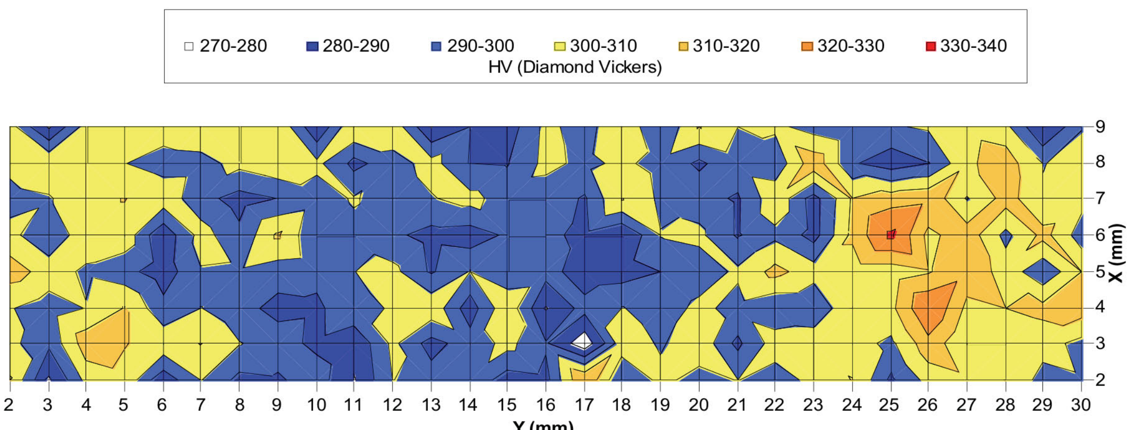




ANL 26%CR plate shows the highest degree of "permanent" grain boundary damage with the minimum spacing (~1 µm) between voids and cracked carbides.

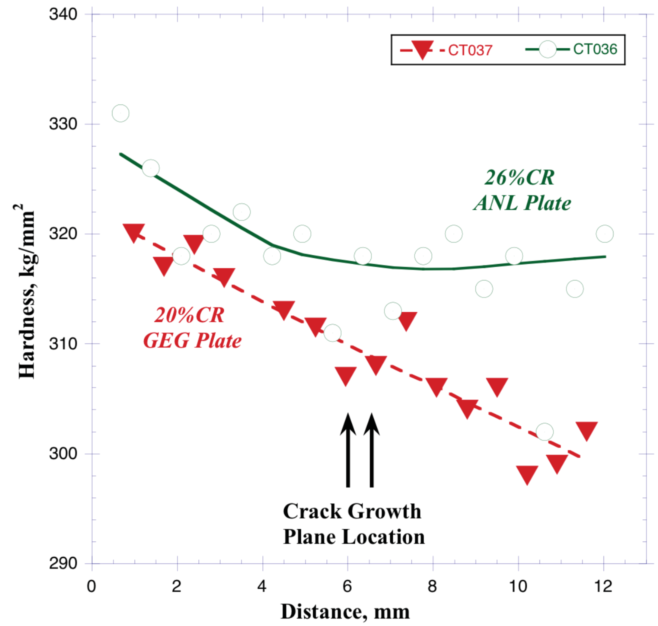
Hardness Measurements for CR Alloy 690 Plate Materials





ANL 26%CR Plate

Considerable variability in thru-thickness hardness for CR plates. Important to establish hardness at SCC crack growth plane.



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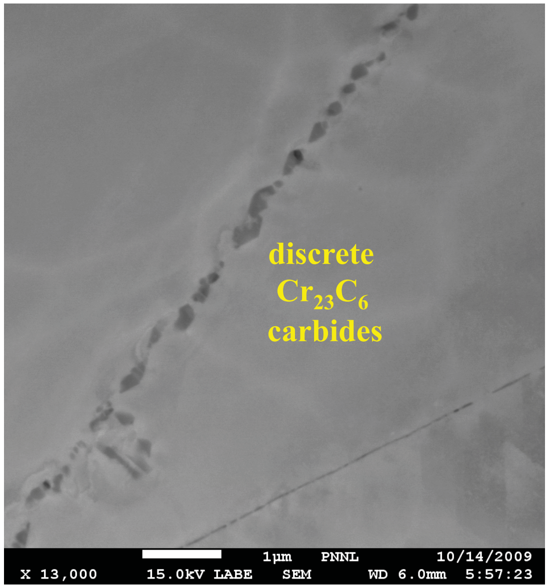
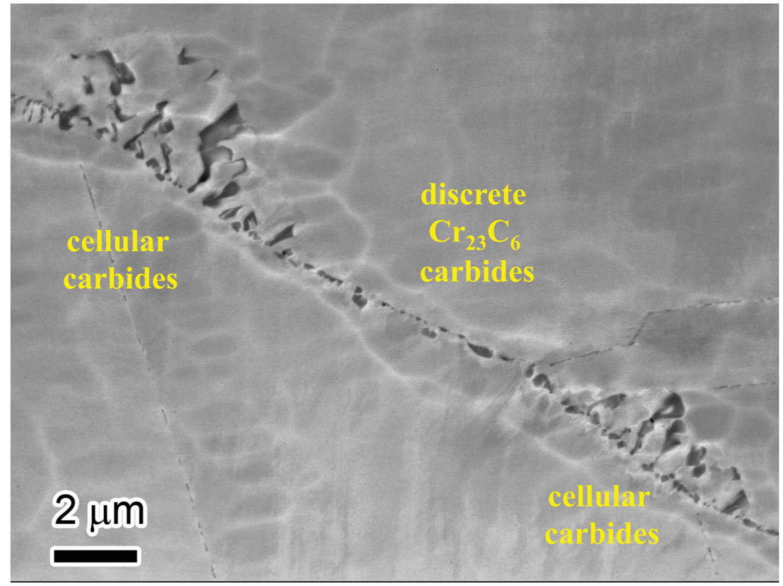
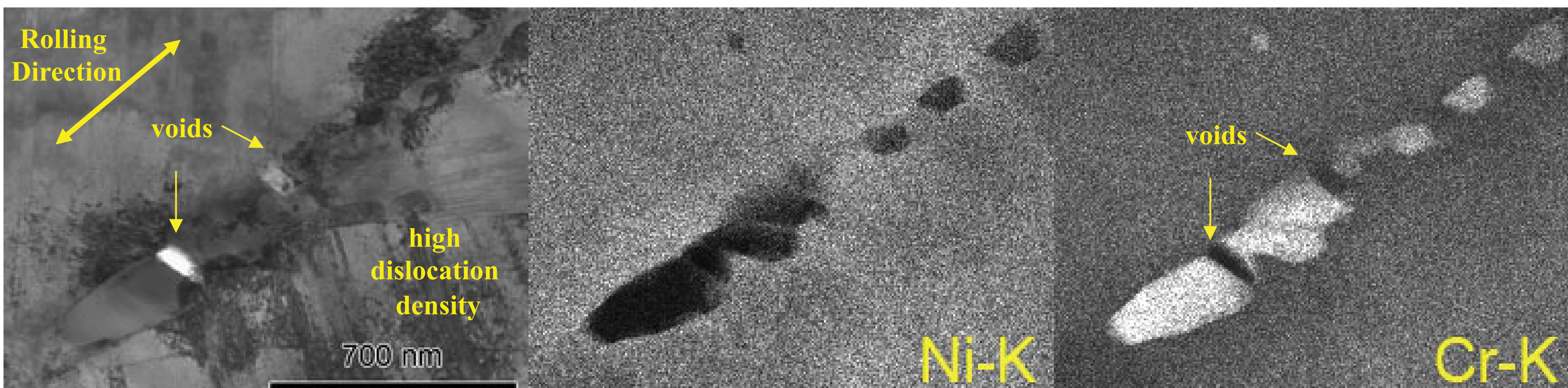
- ***SCC Crack and Crack-Tip Exams on Alloy 690***
  - *SCC morphology and crack path interactions with CR damage*

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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Microstructure Characterizations for CR CRDM Alloy 690TT Heat RE243

Alloy 690 CRDM Heat RE243: As-received, TT condition + 17%CR

Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Equiaxed grains, avg. size ~100 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Depletion	TEM-EDS	Cr minimum ~21-24 wt%, width 300-400 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	250 kg/mm^2 (CT crack plane)
Damage µS	SEM/TEM	High dislocation density, few GB voids, few cracked GB $M_{23}C_6$, and matrix/GB TiN
Strain Dist.	EBSD	Moderate in matrix increasing at GBs

Alloy 690 CRDM Heat RE243: As-received, TT condition + 31%CR

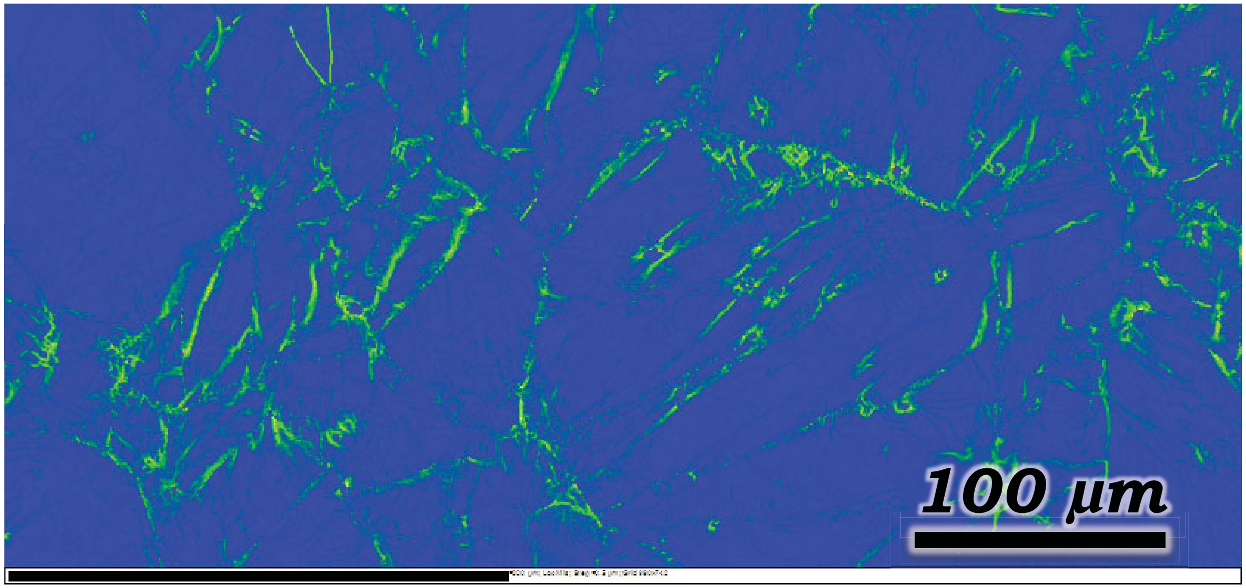
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Slightly elongated grains, avg. size ~100 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Depletion	TEM-EDS	Cr minimum ~21-24 wt%, width 300-400 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	300 kg/mm^2 (CT crack plane)
Damage µS	SEM/TEM	High dislocation density, many GB voids and some cracked GB $M_{23}C_6$, and matrix TiN
Strain Dist.	EBSD	High strain levels in matrix increasing at GBs

Effect of Cold Rolling on EBSD-Indicated Strain

Strain is linearly proportional to misorientation parameter

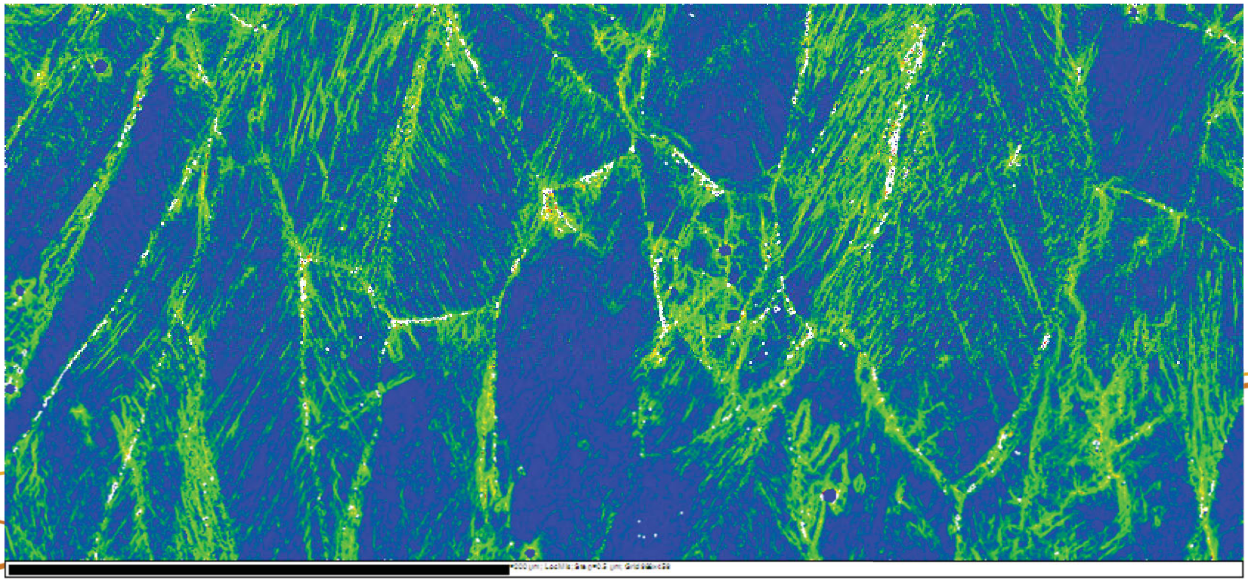
Misorientation represented by green intensity

17%CR
Alloy 690TT
250 kg/mm²



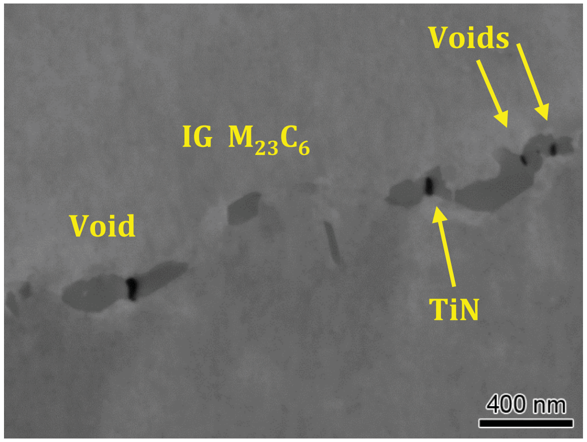
- *Significant difference in strains between 17% and 31%CR.*
- *Strains observed to develop first at GBs.*

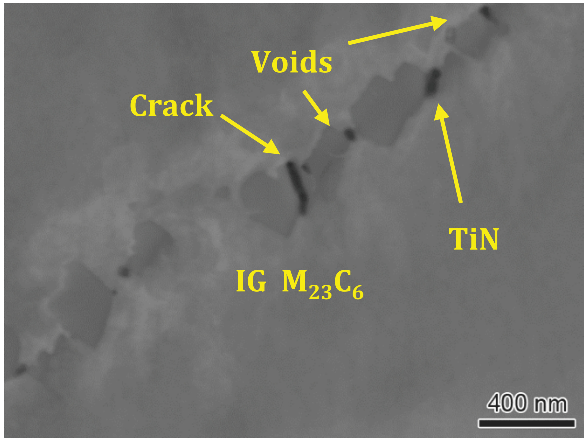
31%CR
Alloy 690TT
300 kg/mm²



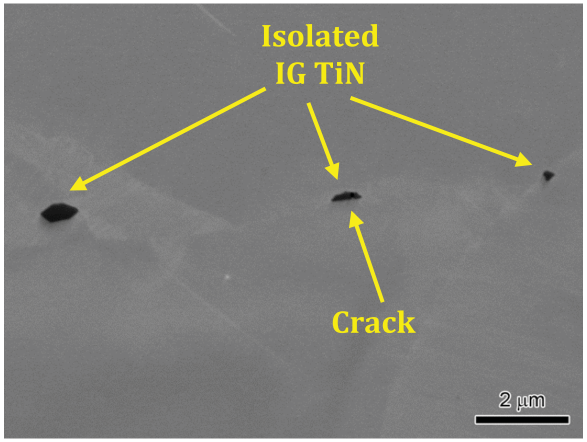
Permanent GB Damage Microstructures in Highly CR Alloy 690TT versus Alloy 690SA

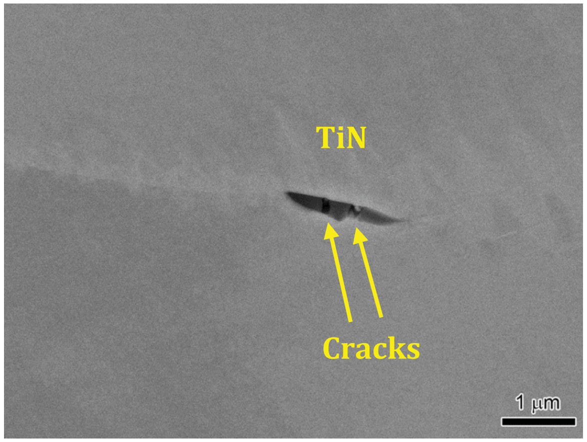
30%CR alloy 690TT CRDM





30%CR alloy 690SA CRDM





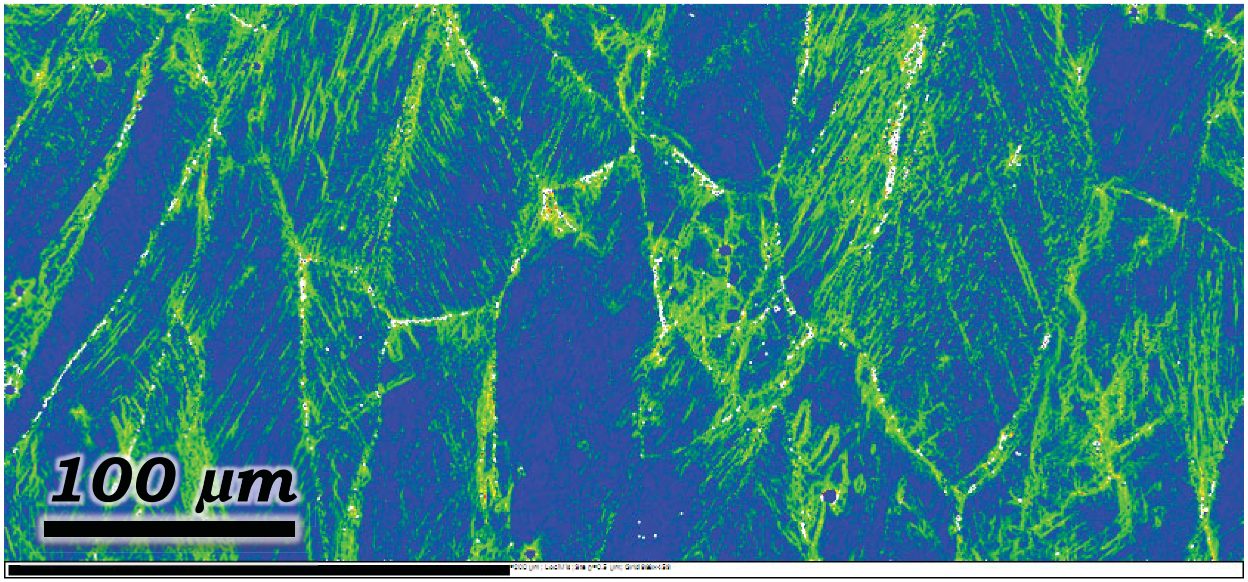
A moderate density of GB voids and some cracked carbides present in 30-31%CR alloy 690TT materials versus <u>isolated</u> observations of cracked GB TiN particles in 30-31%CR alloy 690SA materials.

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

Strain is linearly proportional to misorientation parameter

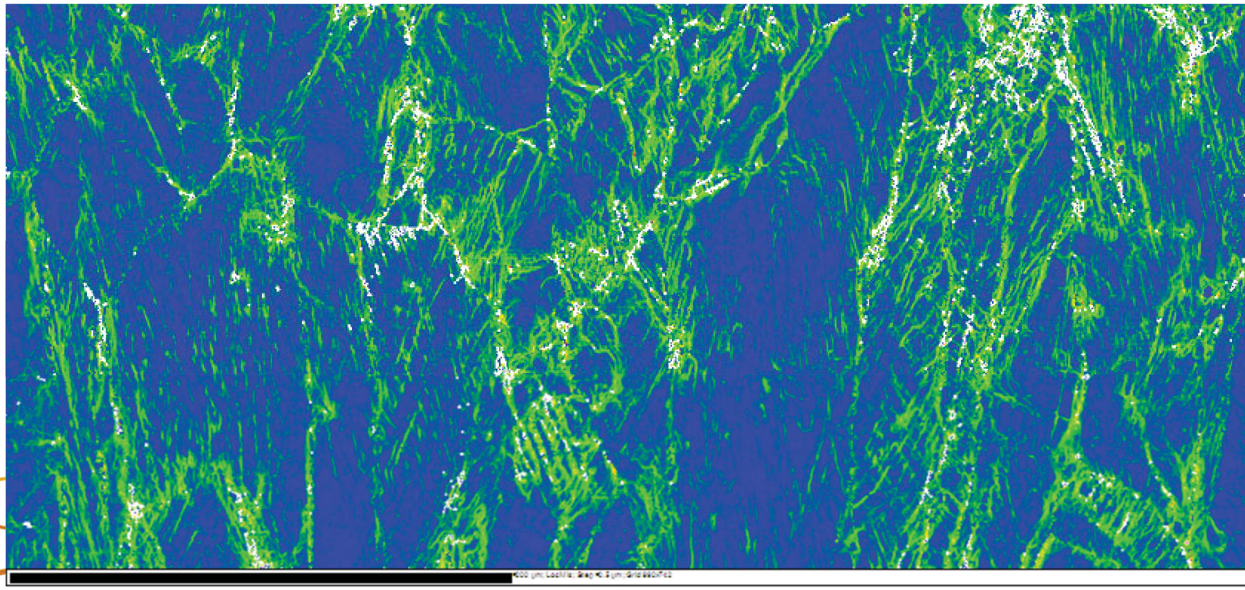
Misorientation represented by green intensity

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



Microstructure Characterizations for CR CRDM Alloy 690TT Heat RE243

Alloy 690 CRDM Heat RE243: As-received, TT condition + 31%CR

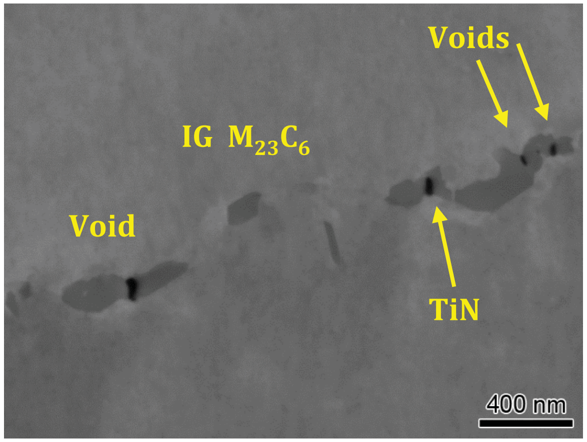
Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Slightly elongated grains, avg. size ~100 µm
IG Precipitates	OM/SEM/TEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Depletion	TEM-EDS	Cr minimum ~21-24 wt%, width 300-400 nm
TG Precipitates	OM/SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	300 kg/mm^2 (CT crack plane)
Damage µS	SEM/TEM	High dislocation density, many GB voids and some cracked GB $M_{23}C_6$, and matrix TiN
Strain Dist.	EBSD	High strain levels in matrix increasing at GBs

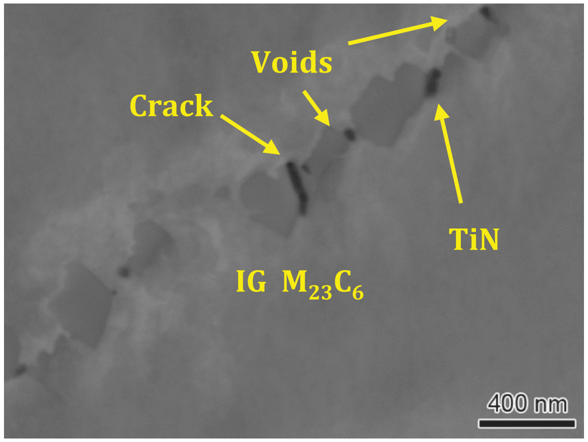
Alloy 690 CRDM Heat RE243: As-received, TT condition + 31%CR + 700C/1h

Microstructural Characteristic	Measurement Technique	Description
Grain Shape/Size	OM	Slightly elongated grains, avg. size ~100 µm
IG Precipitates	SEM	Semi-continuous discrete (100-200 nm) and cellular $M_{23}C_6$, occasional TiN (200-500 nm)
GB Cr Depletion	TEM-EDS	Not measured, assume similar to TT
TG Precipitates	SEM	Few random TiN or Ti carbonitrides (~1 µm)
Hardness	Vickers	~270 kg/mm^2
Damage µS	SEM	High dislocation density, many GB voids and some cracked GB $M_{23}C_6$, and matrix TiN
Strain Dist.	EBSD	Moderate strain level increasing at GBs

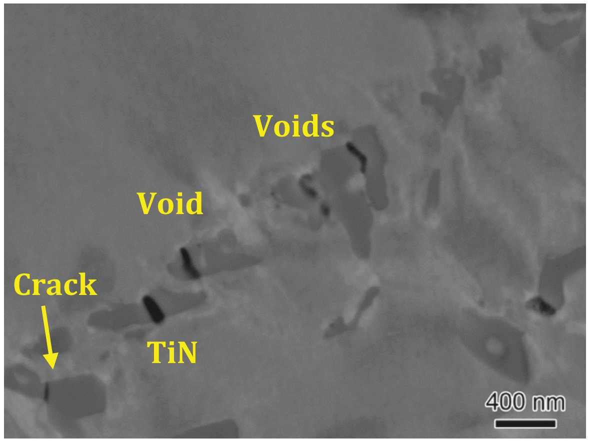
Effect of Recovery Anneal on the Permanent GB Damage Microstructures

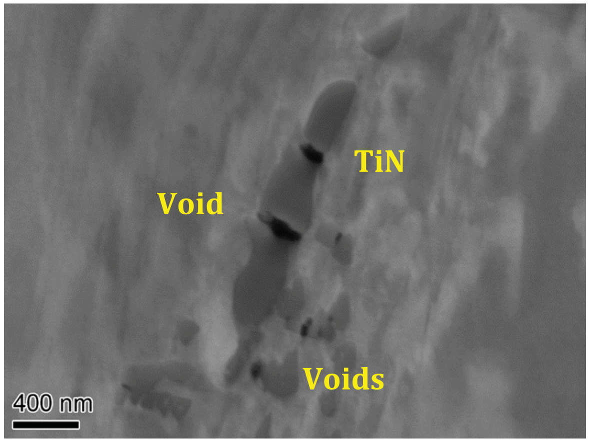
31%CR alloy 690TT CRDM





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^{o}C recovery anneal.

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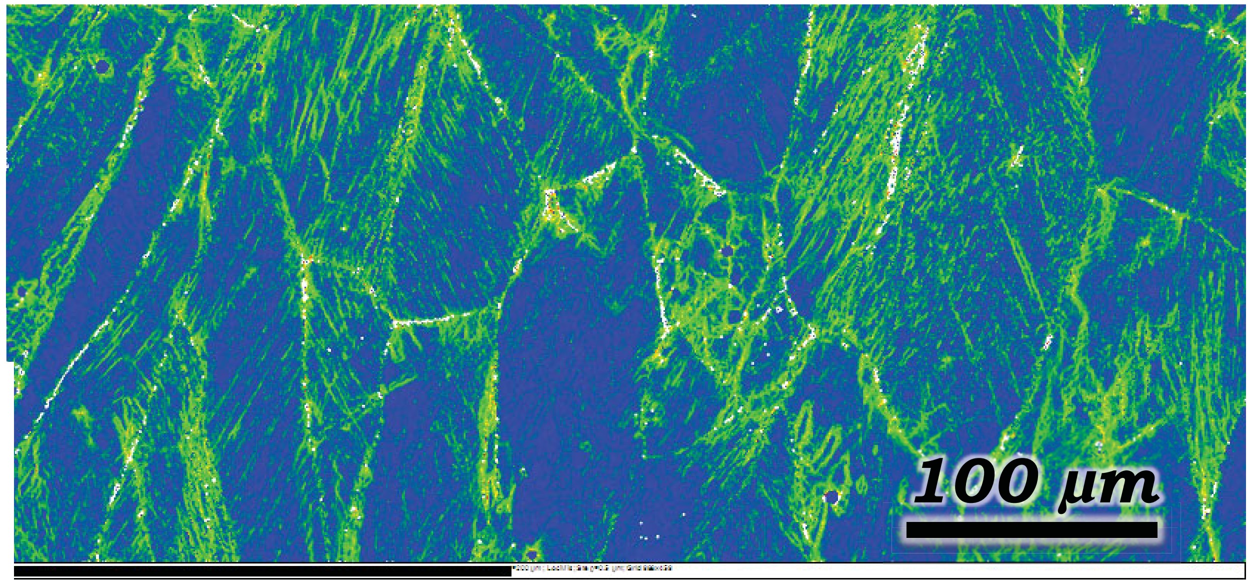
Effect of Recovery Treatment on EBSD-Indicated Strain

Strain is linearly proportional to misorientation parameter

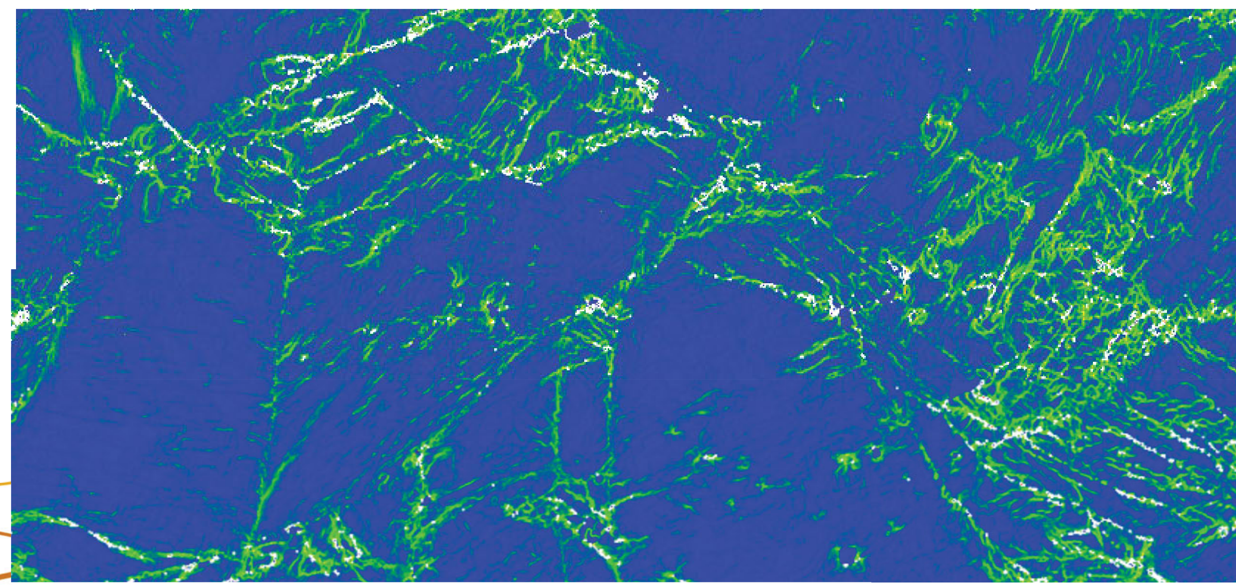
Misorientation represented by green intensity

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.

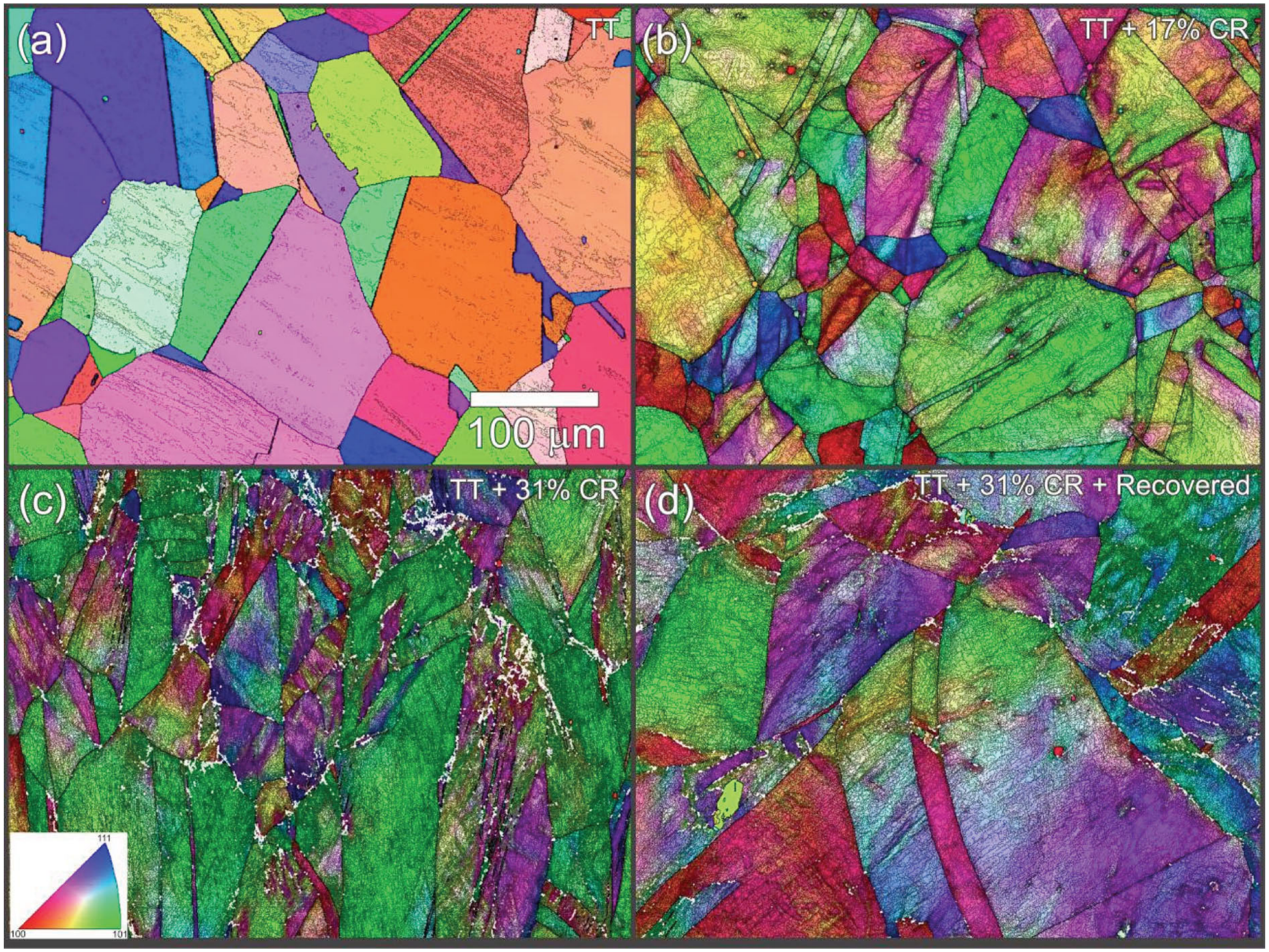
31%CR
Alloy 690TT,
700°C/1 hr/AC
270 kg/mm² →



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

0% CR

17% CR



31% CR

31%CR + Recovery

EBSD TD inverse pole figure maps showing significant change from 17-31%CR and minor effect of recovery anneal.

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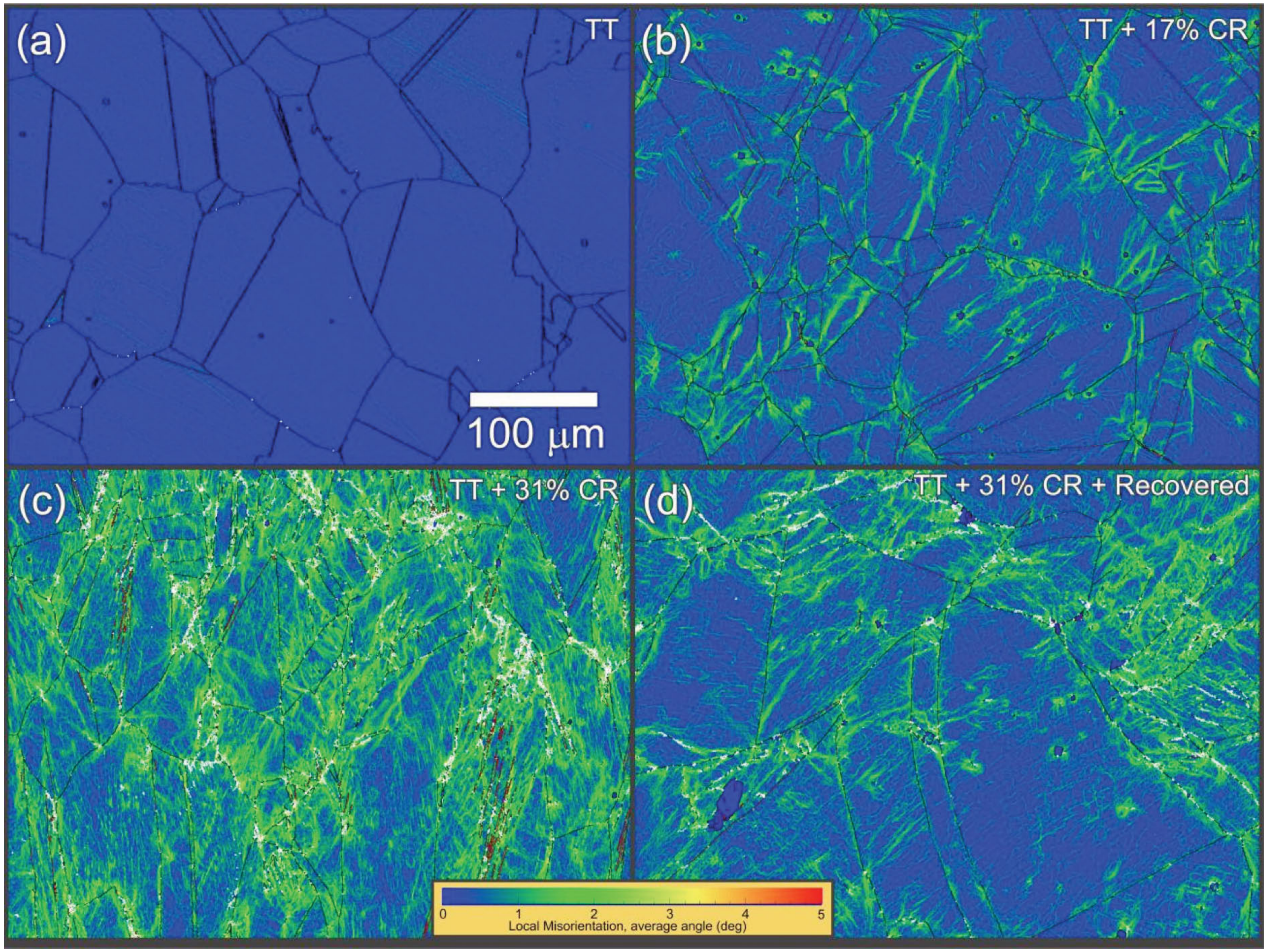
Influence of Cold Rolling on Strain Distributions for Alloy 690TT CRDM Tubing

0% CR

17% CR



31% CR

31%CR + Recovery

Local misorientation maps showing significant change from 17-31%CR and minor effect of recovery anneal.

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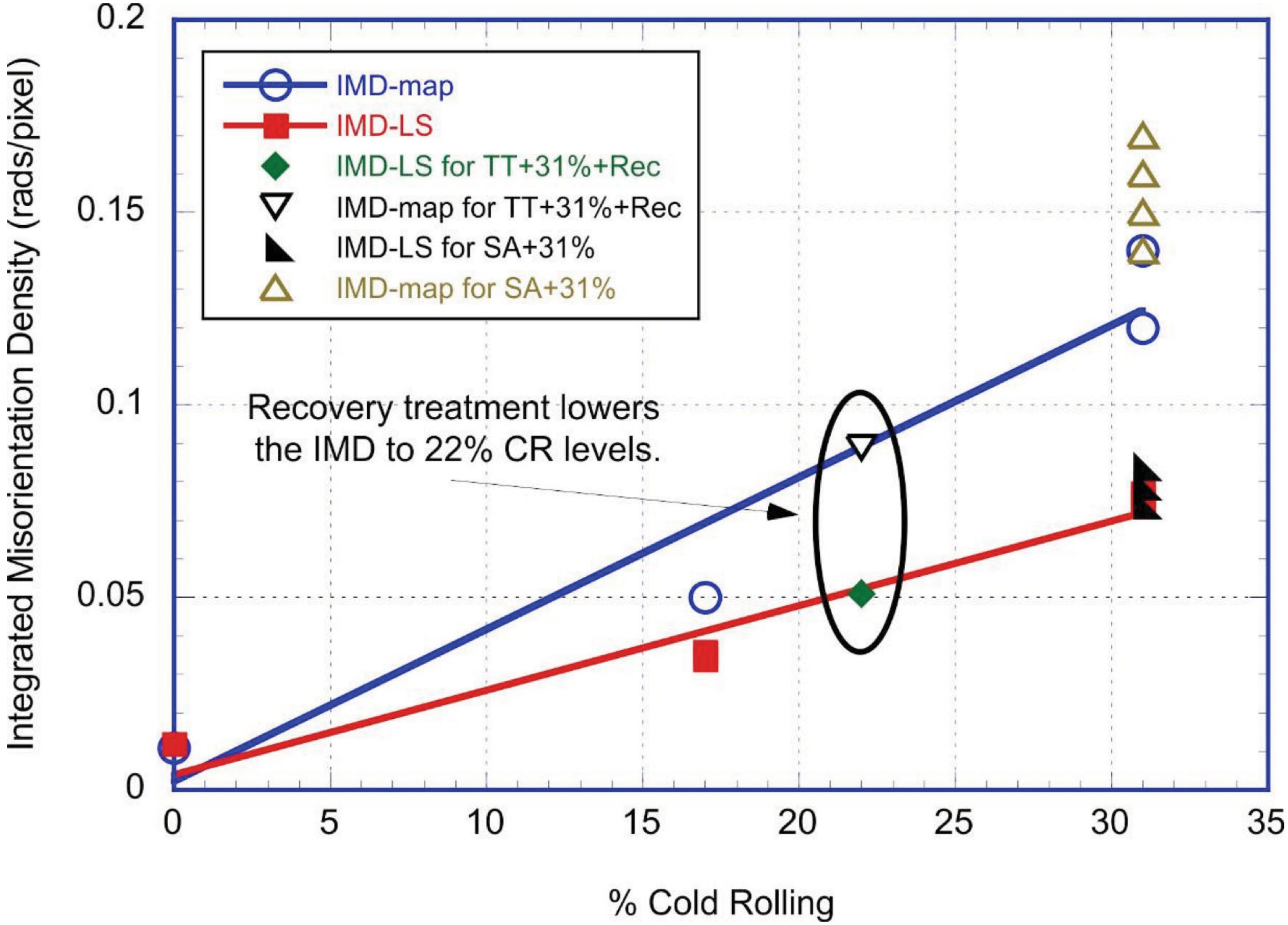
Influence of Cold Rolling on Strain Distributions for Alloy 690TT CRDM Tubing



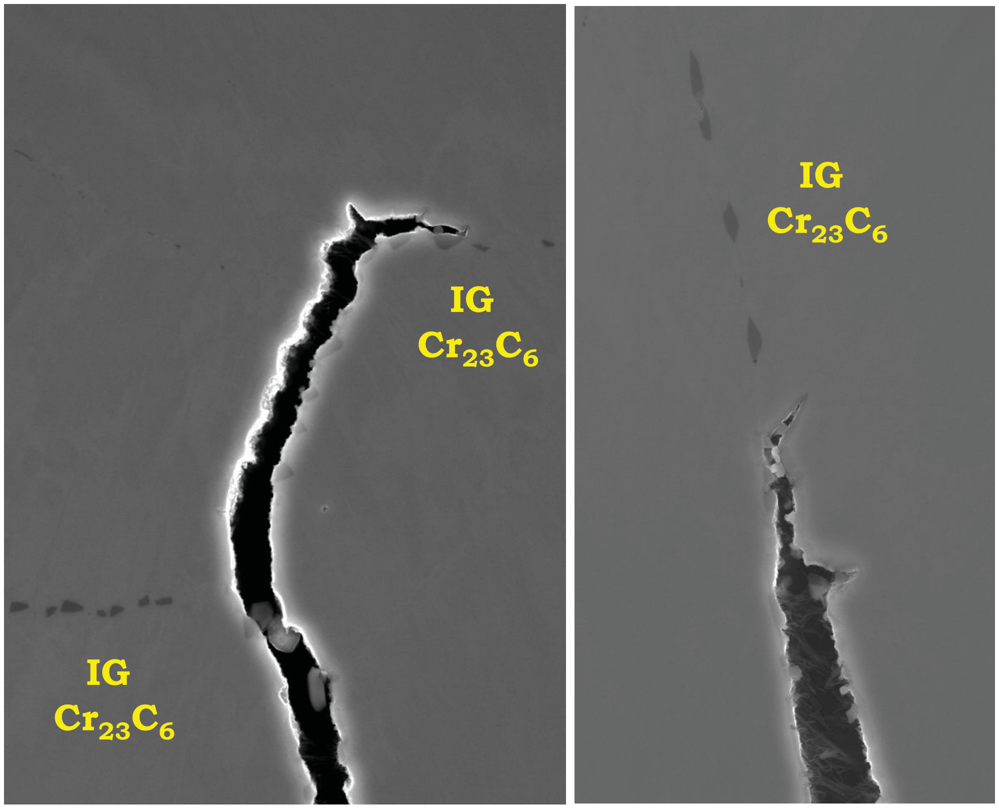
Data from line scans and maps are included, and both indicate that a linear relationship can be inferred between the integrated misorientation density (IMD) and increasing levels of cold rolling .

PNNL Characterization Actvities: Examples for Alloy 690 Materials

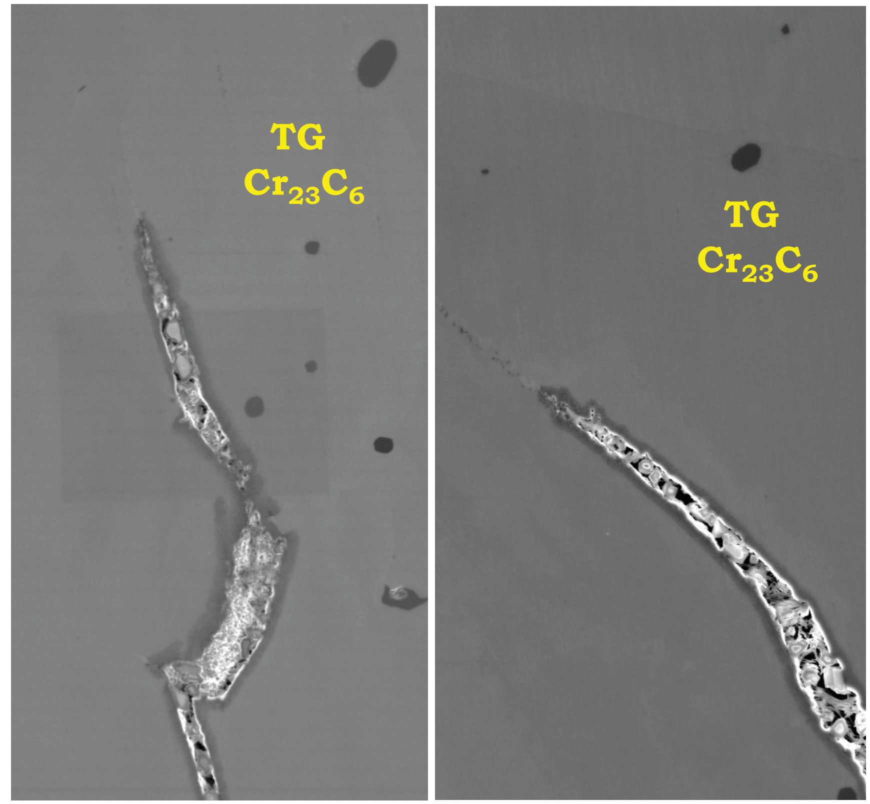
- ***Initial Alloy 690 Microstructures***
  - *As-received thermally treated (TT), solution annealed (SA) or desensitized alloy 690 CRDM tubing*
  - *As-received alloy 690 mill annealed (MA) plate heats*
- ***Cold Work Effects on Alloy 690 Microstructures***
  - *26%CR ANL (NX3297HK12) and 20%CR GEG (B25K) heats*
  - *17-31%CR CRDM alloy 690TT and alloy 690SA, influence of recovery anneal after 31%CR alloy 690TT (heat RE243)*
- ***SCC Crack and Crack-Tip Exams on Alloy 690***
  - *SCC morphology and crack path interactions with CR damage*

SCC Crack Tips in Alloy 690 CR Plates

CT036 - ANL 26%CR Heat



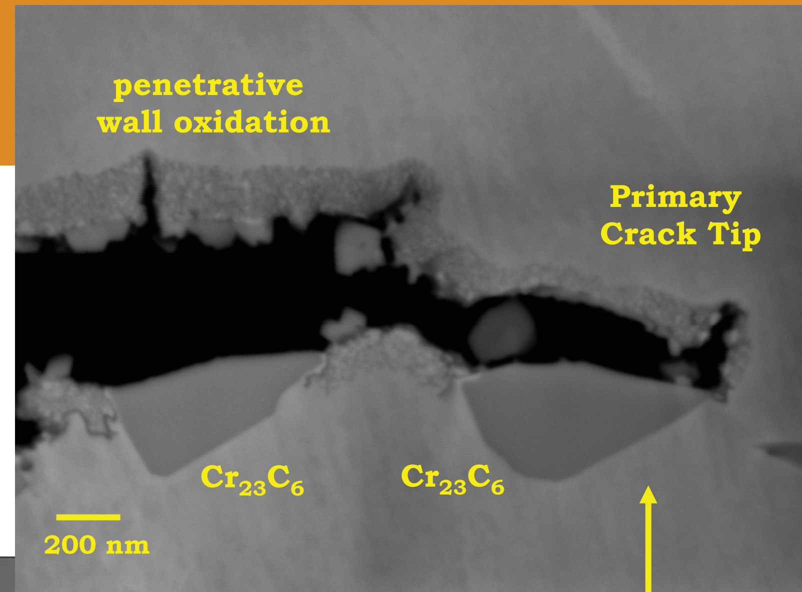
CT037 - GEG 20%CR Heat

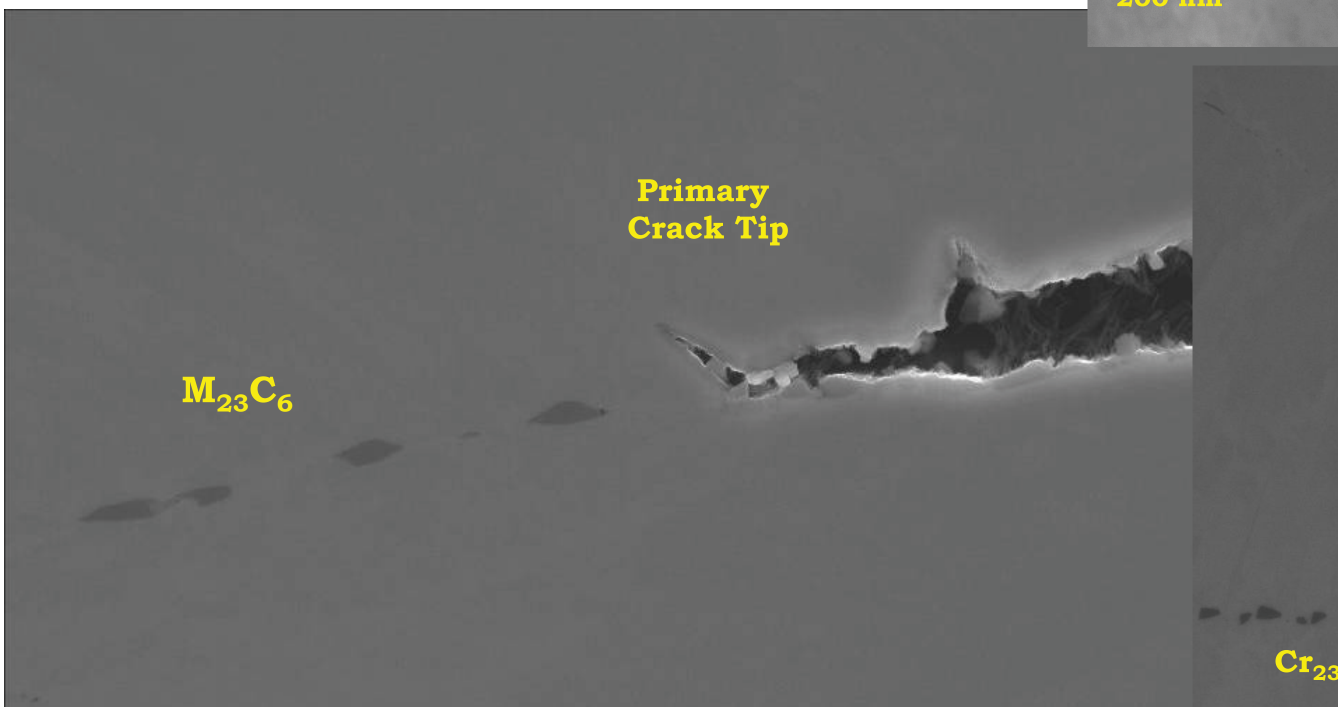


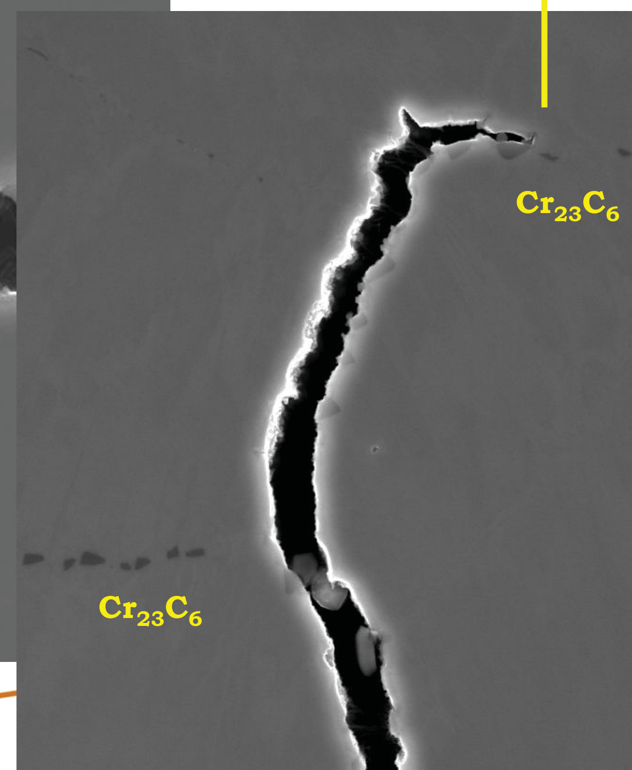
IGSCC in both cases with semi-continuous GB carbides for the ANL sample and only isolated GB carbides for the GEG material. Cracks are open to tips, but contain crystallites and needle-shaped filaments as fill material.

SEM Images at SCC Tips in ANL 26%CR Alloy 690

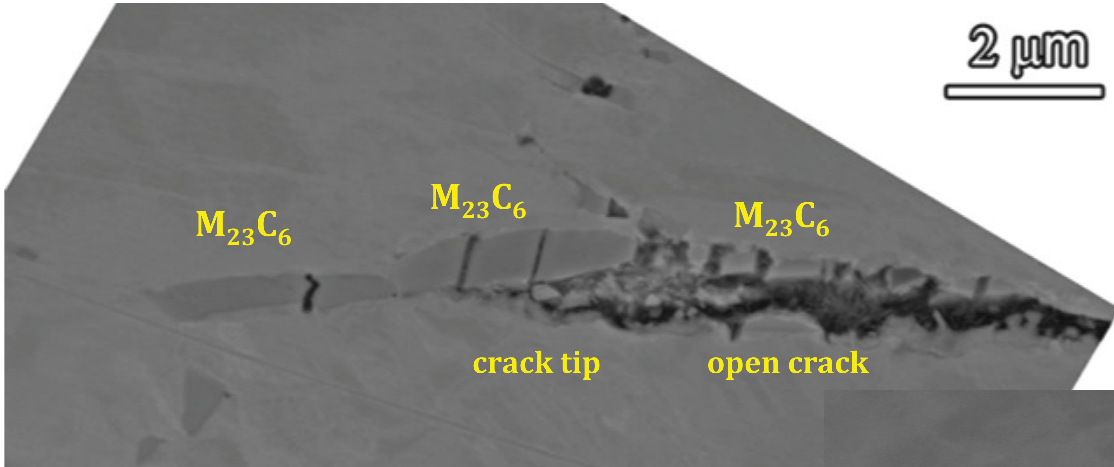
SCC propagates along grain boundaries and carbide/boundary interfaces, no evidence for enhanced crack/void formation ahead of crack tips. Tips often blunt/branched, ending near carbides.





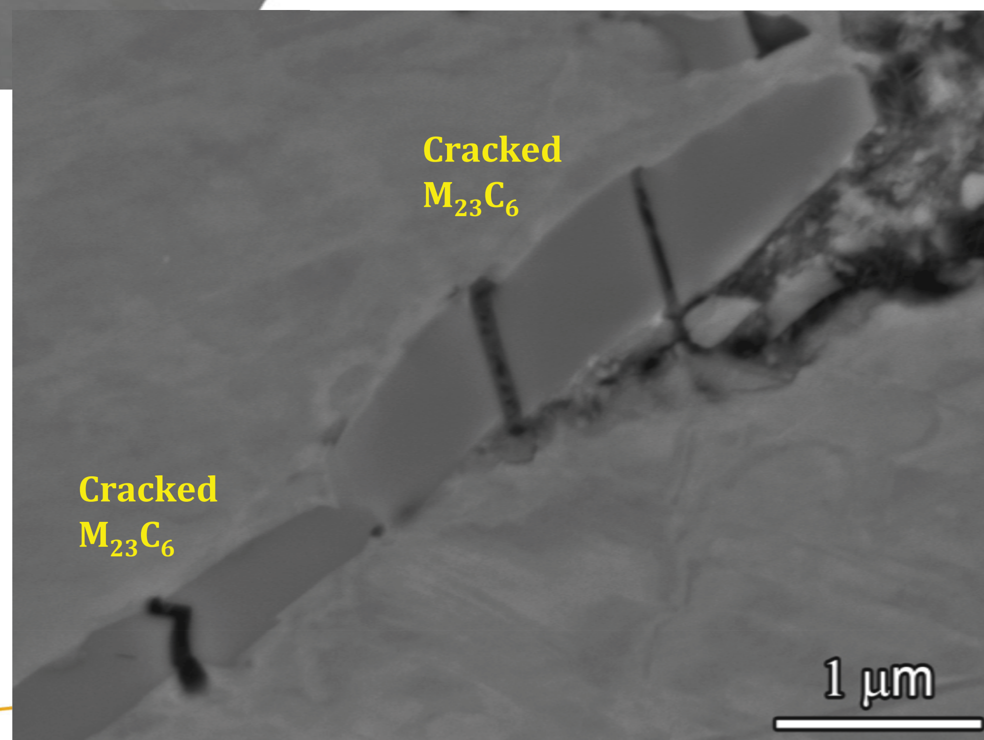


SCC Tip in ANL 26%CR Alloy 690 Plate



Interaction of IGSCC crack with preexisting cracked carbides in the ANL 26%CR alloy 690 material.

SCC propagates along grain boundaries and carbide/ boundary interfaces, no evidence for enhanced crack/ void formation ahead of crack tips. Tips often blunt/ branched, ending near carbides.



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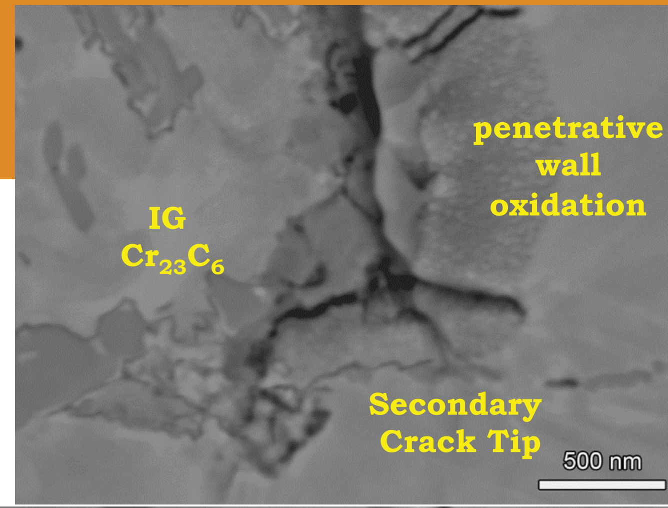
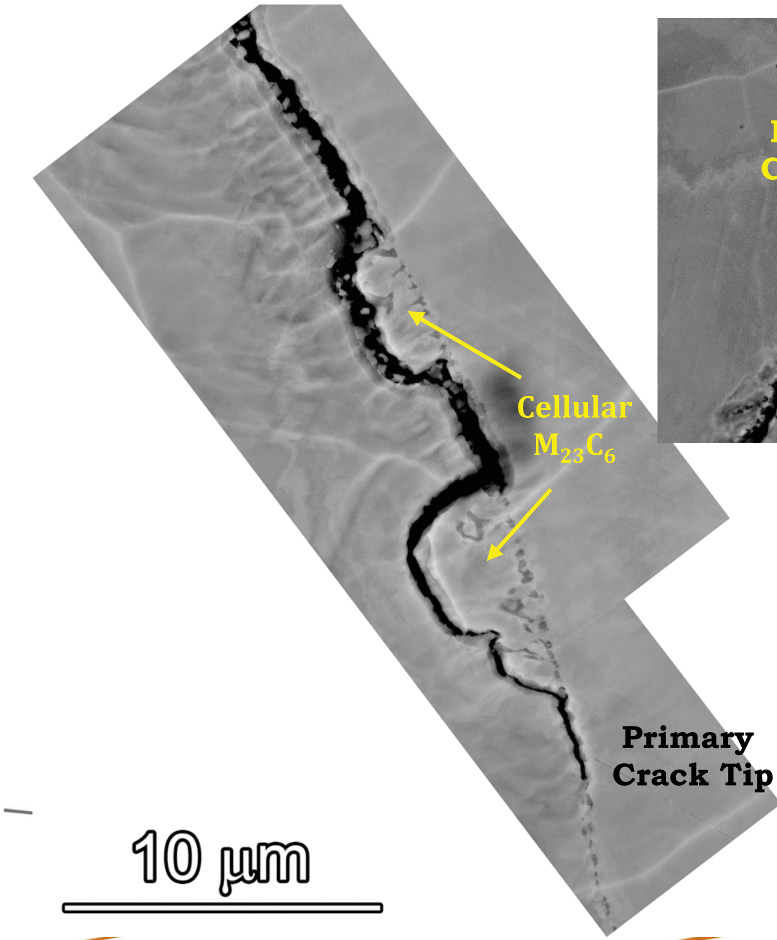
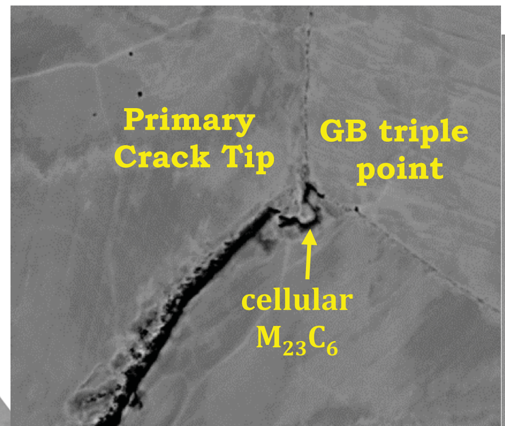
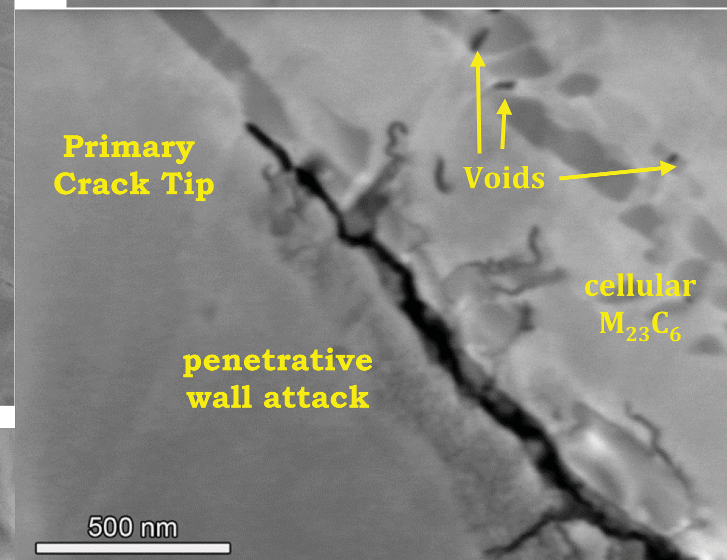
SEM Images at SCC Crack Tips in 30%CR Alloy 690 CRDM Tube

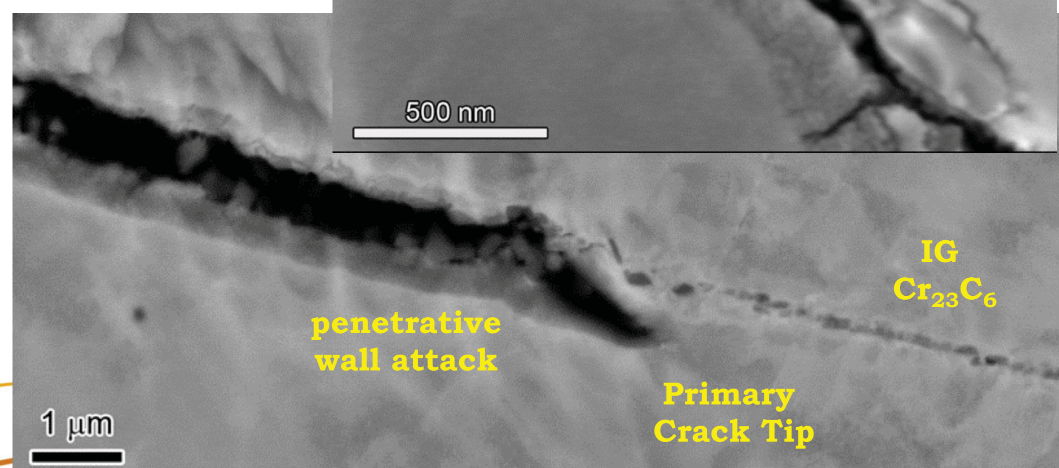
SCC propagates along grain boundaries and carbide/boundary interfaces, follows migrated boundaries around cellular carbides in 30%CR CRDM material. No evidence for enhanced crack/void formation ahead of crack tips.





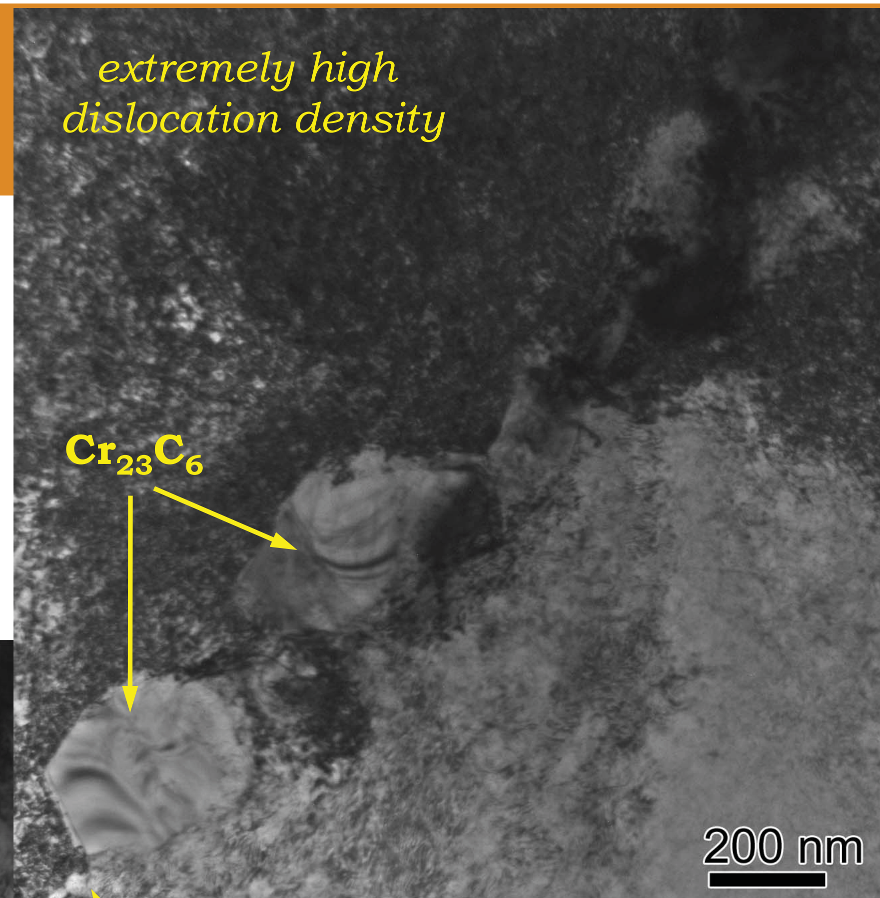


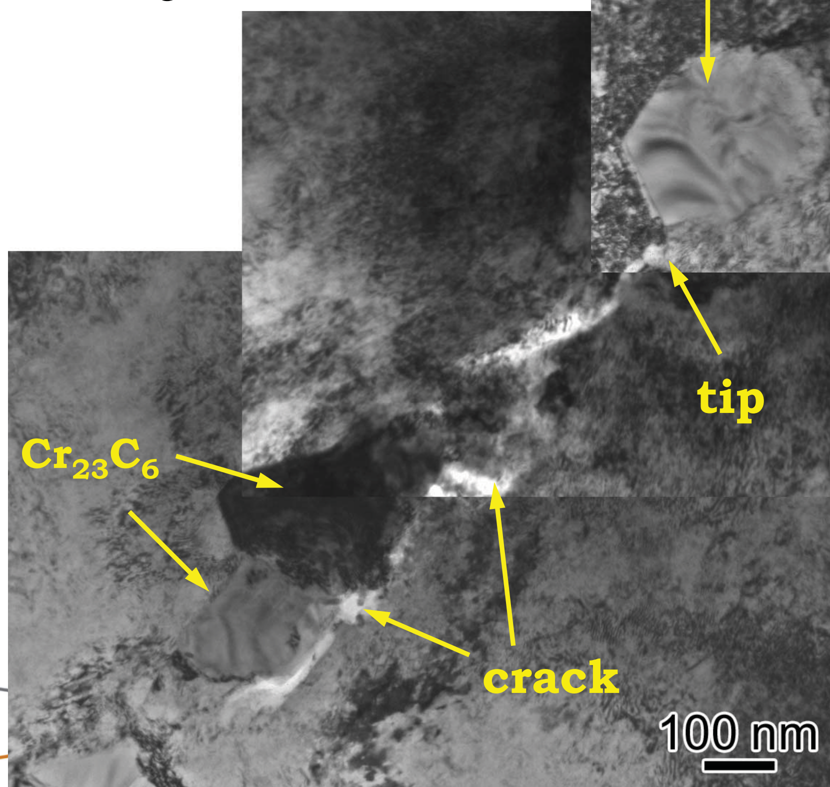




IGSCC Crack Tip in 30%CR Alloy 690TT

Grain boundary characteristics have been examined ahead of IGSCC crack tips after further ion milling. Extremely high dislocation densities are present along with isolated voids from initial cold rolling.





No evidence has been found for ***enhanced*** *void or crack formation ahead of the oxidized IGSCC tips.*

PNNL Characterization Actvities: Examples for Alloy 690 Materials

Microstructural Characterization

- *Essential for material assessment and comparisons including heat-to-heat, processing and heat treatment effects.*
- *Important to assess general microstructure (grain size/ shape, banding), precipitate microstructures (size/ distribution IG and TG), local microchemistry (grain boundary depletion/ segregation), matrix hardness and strain distributions.*
- *Open question how detailed characterizations should be on most materials, depends on specific issues being examined.*

Characterization Methods

- *Optical metallography, SEM and EBSD for general microstructure*
- *SEM and TEM for precipitate microstructure*
- *TEM for grain boundary microchemistry and phase identification*
- *EBSD for strain distributions*
- *Optical, SEM and TEM of SCC cracks and crack tips*