Microstructural Effects on Stress Corrosion Crack Growth in Cold-Worked Alloy 690 Tubing and Plate Materials

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Research Supported by

U.S. Nuclear Regulatory Commission

and Rolls Royce

16th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors

August 2013 Asheville, North Carolina, USA

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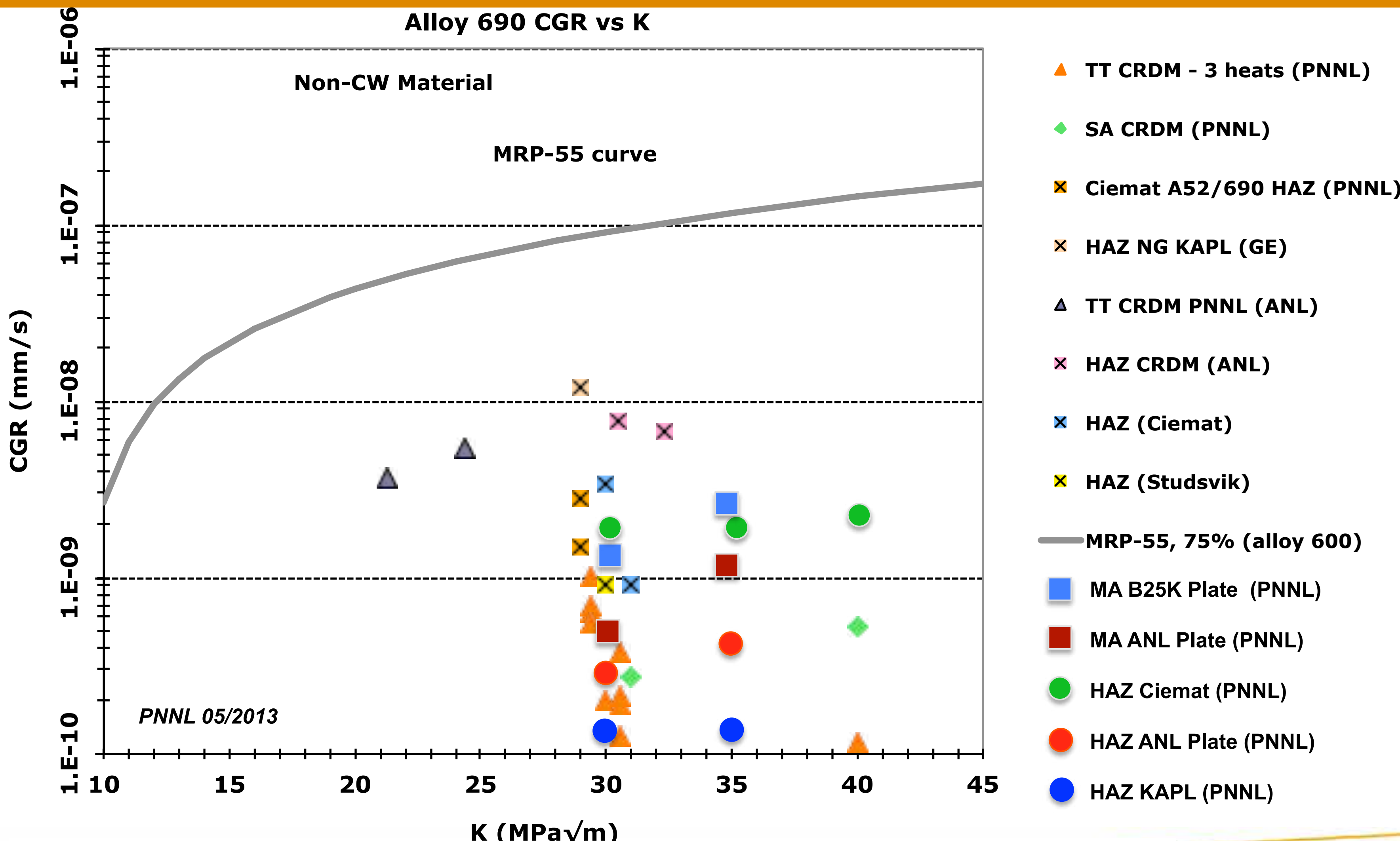
Measured SCC Growth Rates for Alloy 690 Materials without Cold Working



Measured SCC growth rates are low or very low on alloy 690 materials in the non cold-worked condition, however the total number of CRDM tubing and plate heats evaluated is limited.

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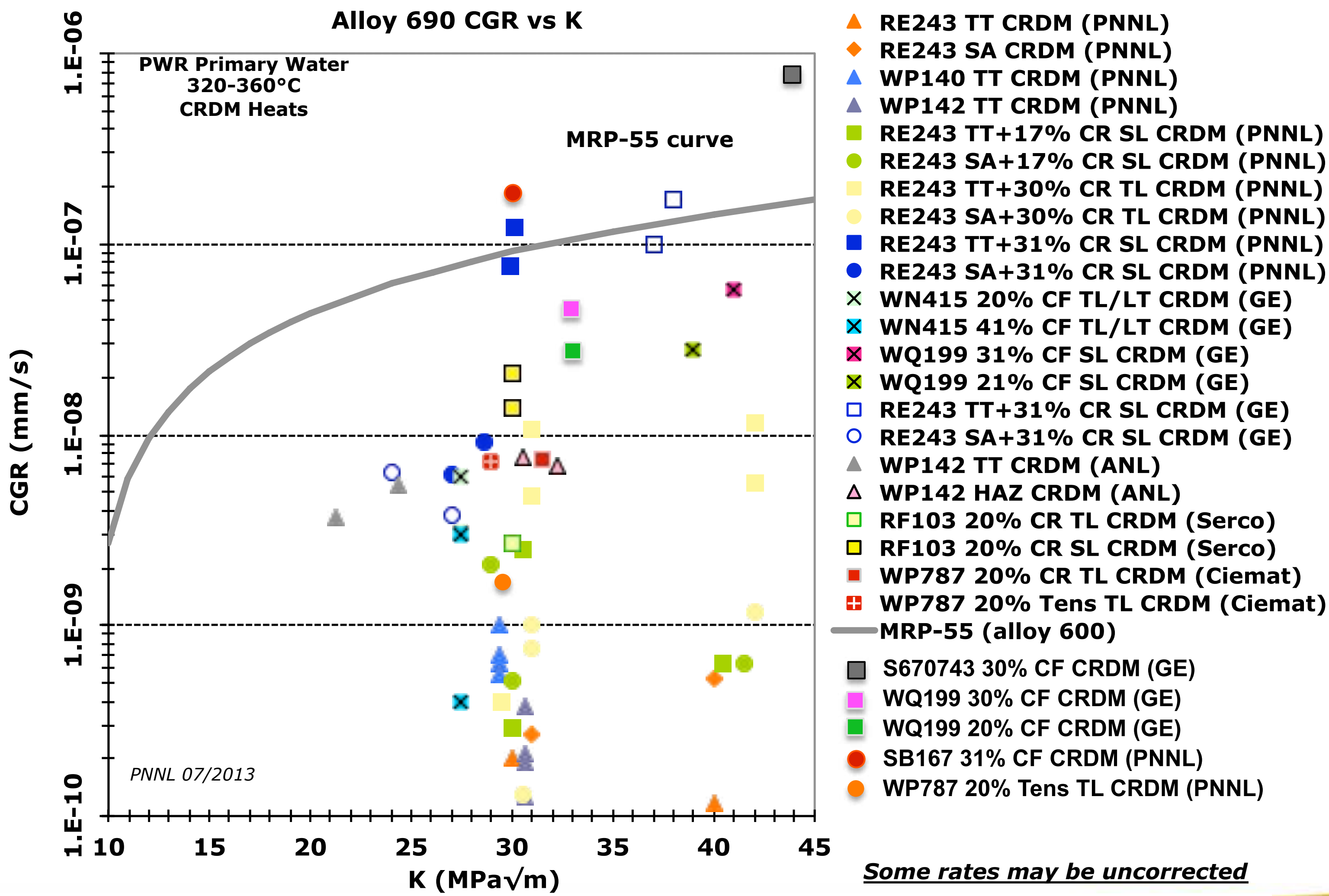
Measured SCC Growth Rates for Alloy 690 CRDM Tubing Materials



Some rates may be uncorrected

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 (≥~20%) can promote moderate-to-high propagation rates.

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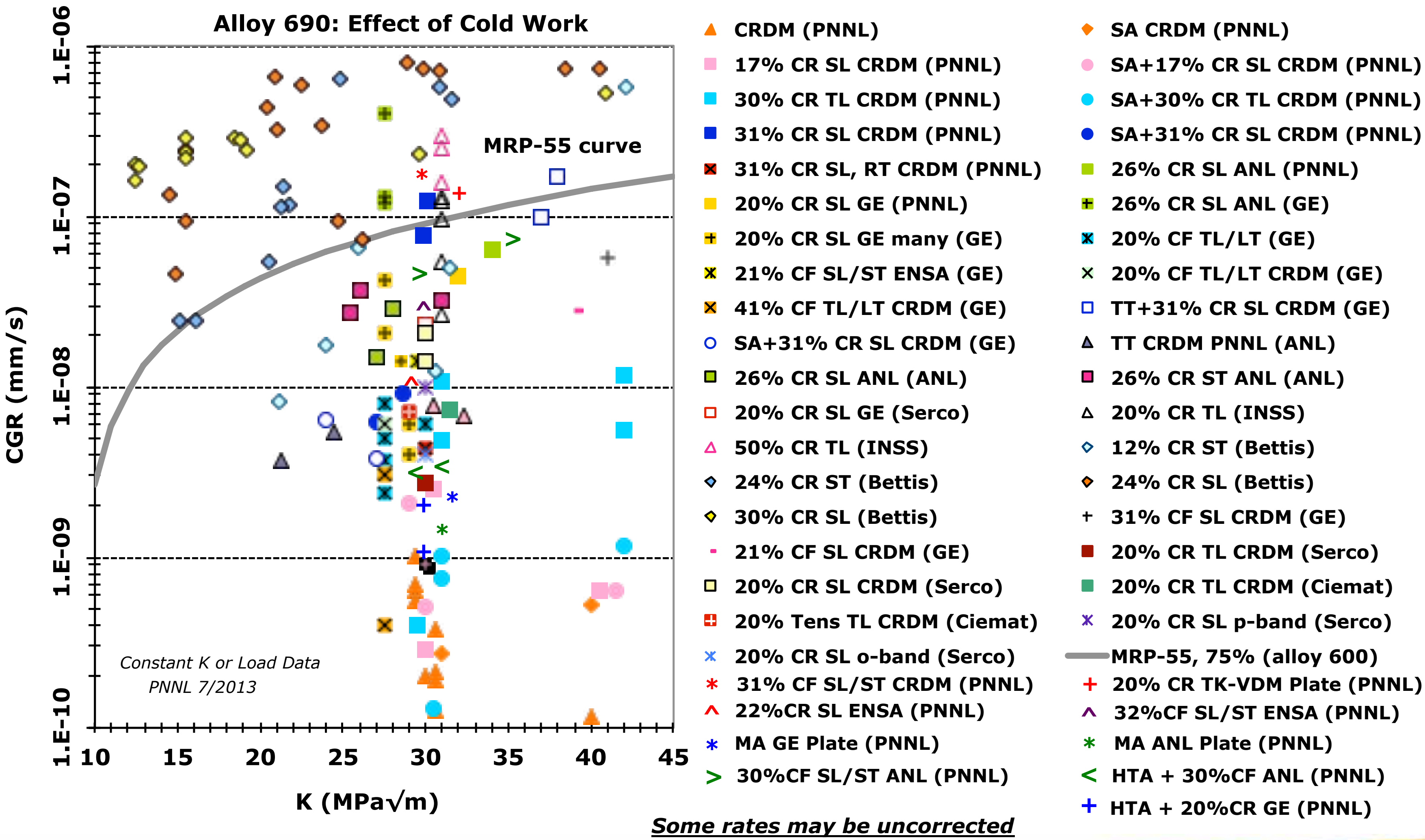
Summary of Alloy 690 Measurements of SCC Growth Rates – All Data



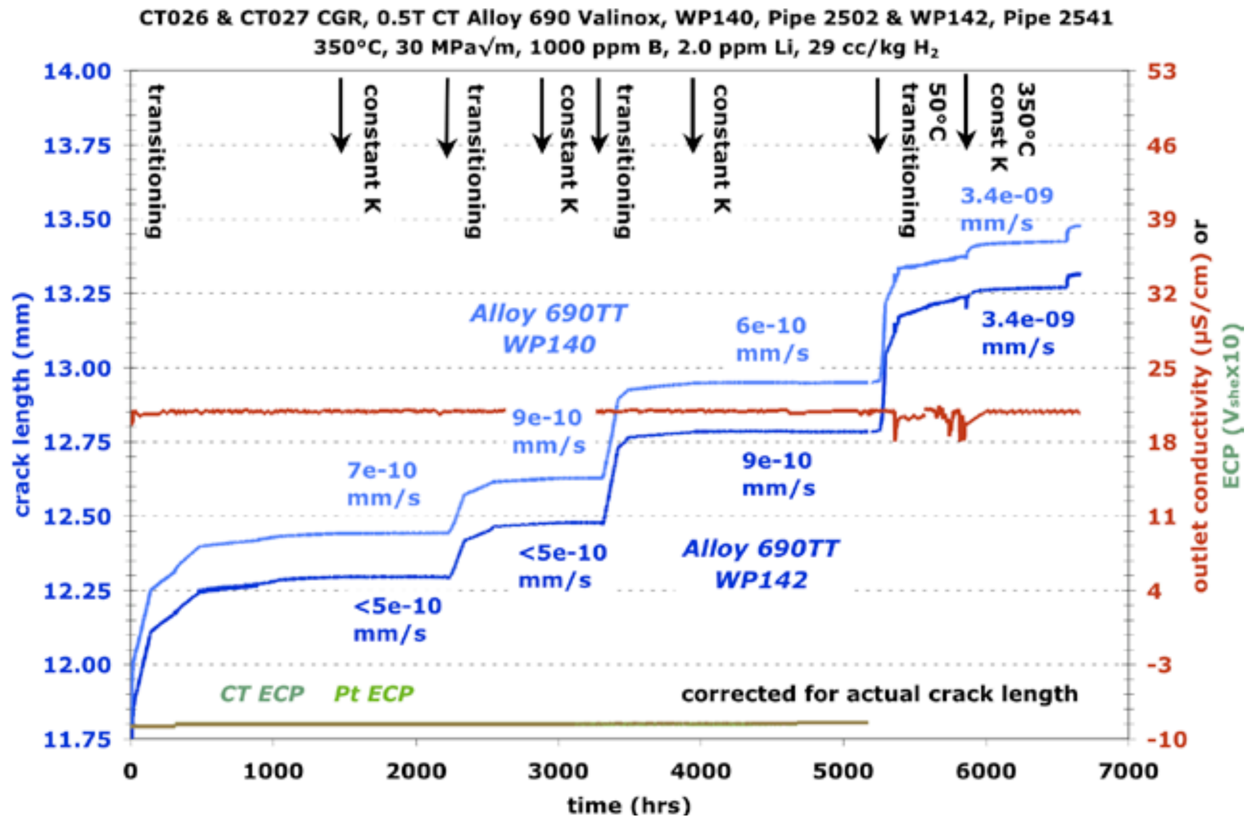
Full spectrum of measured SCC growth rates illustrating significant effect of 1D cold work, however initial Bettis results remain at upper end of data with extremely high growth rates at lower K values.

PNNL Alloy 690 Testing Summary
August 2013

- ***Alloy 690 CRDM Tubing (15 tests)***
  - *Heat-to-heat response with tests on three as-received TT heats: Valinox RE243, WP140 and WP142*
  - *As-received TT (high density of GB Cr carbides + Cr depletion) versus HTA/SA (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: 31%CR alloy 690TT + 700°C (S-L)*
  - *As-received TT + 21%CF & 30%CF Valinox RE243, 30%CF Sumitomo SB167 and 20%TS Valinox (from CIEMAT)*
- ***Alloy 690 Plate Materials (10 tests)***
  - *As-received MA heats: ANL (NX3297HK12) and GEG (B25K)*
  - *MA versus HTA (most carbides removed) for cold-worked 30%CF ANL (S-L/S-T) and 20%CR (S-L) GEG heats*
  - *26%CR ANL (S-L), 22%CR (S-L) ENSA, 30%CF (S-L/S-T) ENSA and 20%CF TK-VDM*

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.

PNNL Alloy 690 Characterization Activities

- ***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, strain distributions and local CW damage characteristics.*
- ***Characterization Methods***
  - ***Optical, SEM and EBSD for general microstructure***
  - ***SEM and TEM for precipitate and CW damage microstructure***
  - ***EBSD for strain distributions, hardness***
  - *TEM for phase identification and grain boundary composition*
  - *APT for grain boundary composition*
  - *Optical, SEM, TEM and APT of SCC cracks and crack tips*

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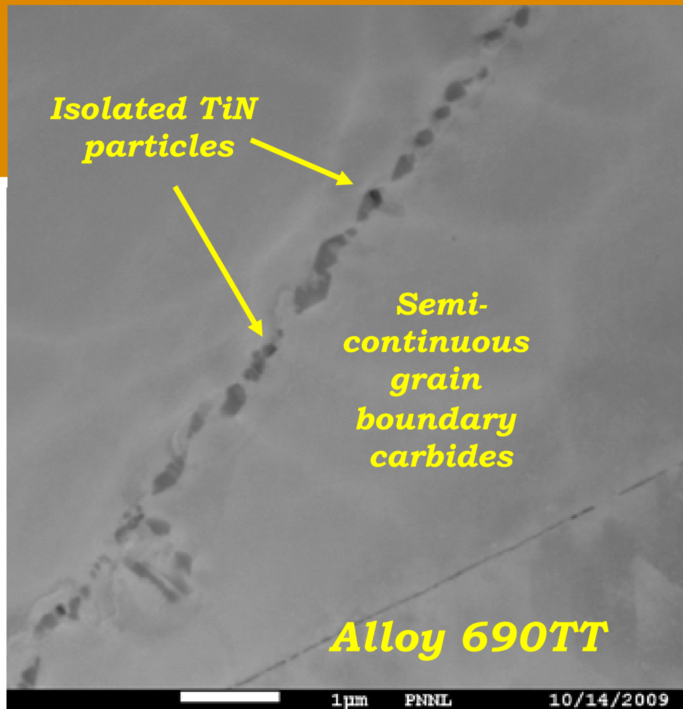
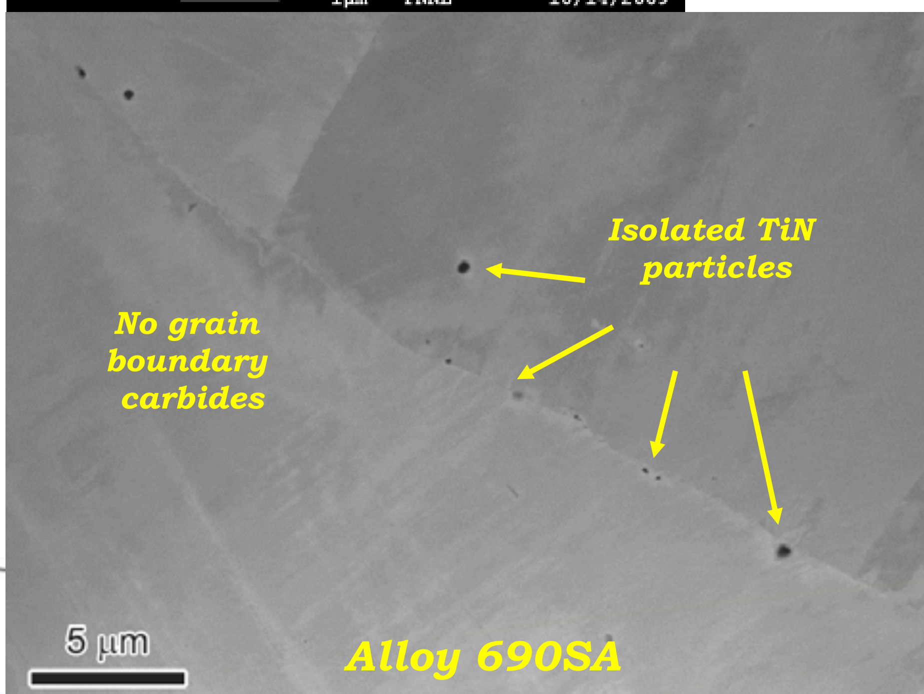
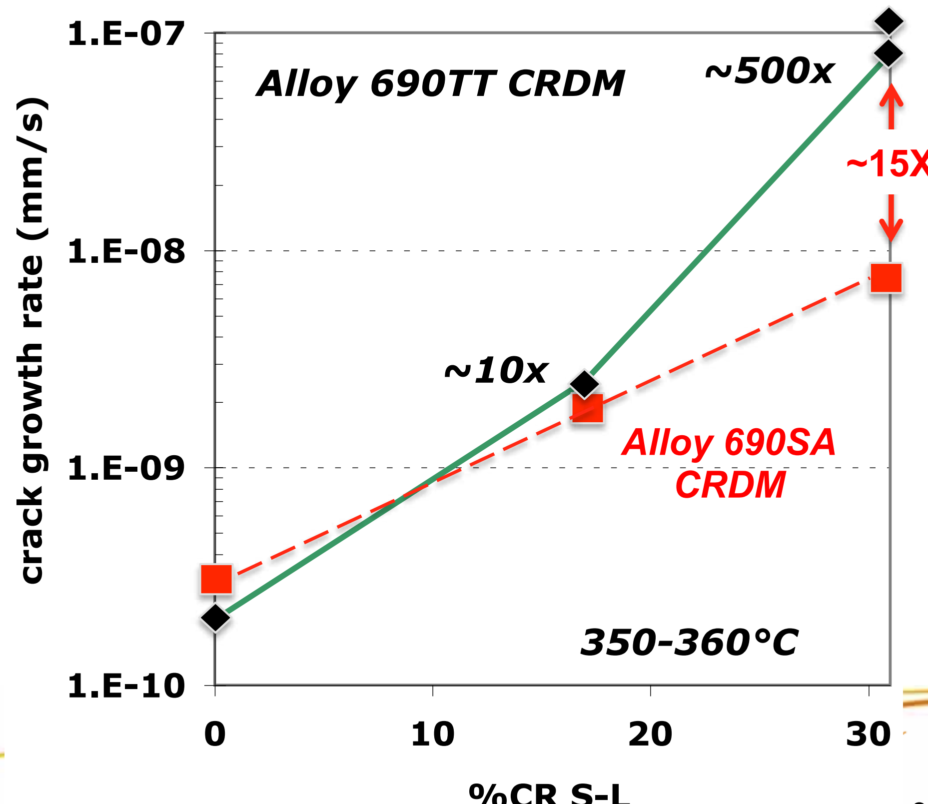
CRDM Alloy 690TT vs Alloy 690SA



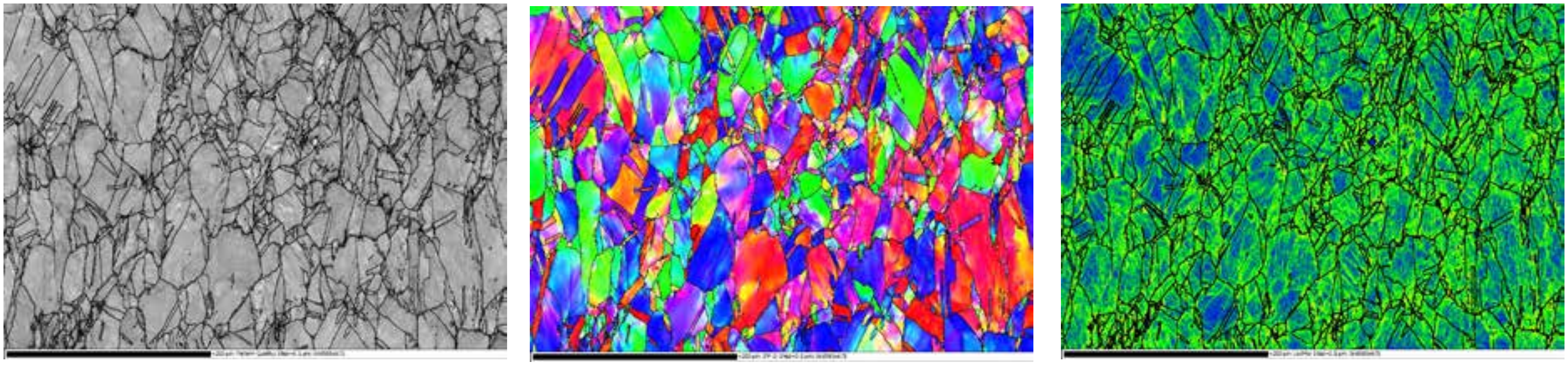


Solution anneal at 1100°C and water quench removed nearly all grain boundary carbides, isolated TiN particles remain. This material was then cold rolled to 17, 30 and 31% as for the alloy 690TT.

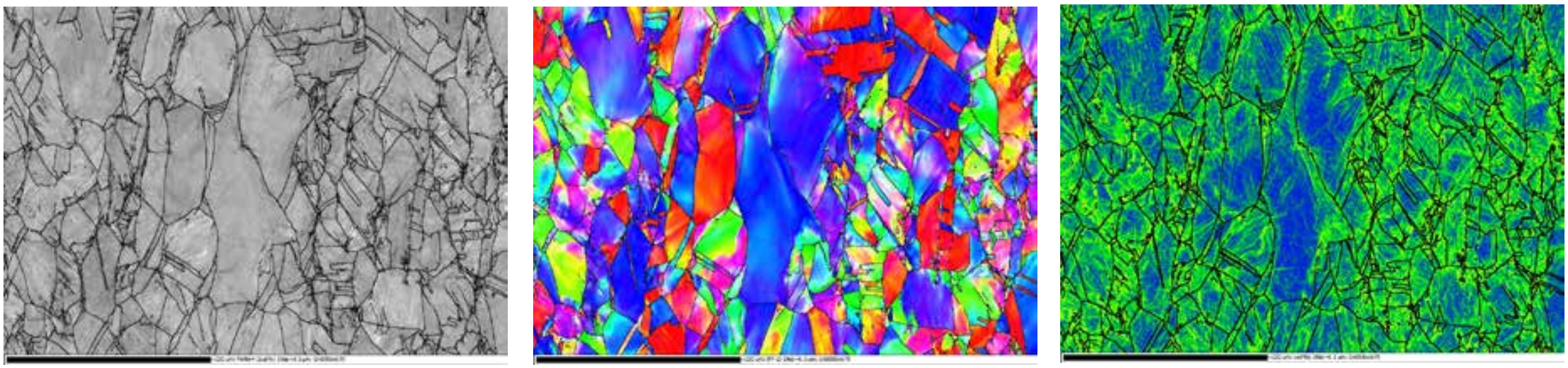


Microstructure and Strain Distributions in ANL MA versus HTA 30%CF Alloy 690 Plate

ANL MA + 30%CF: Average Vickers Hardness = 316 kg/mm^2

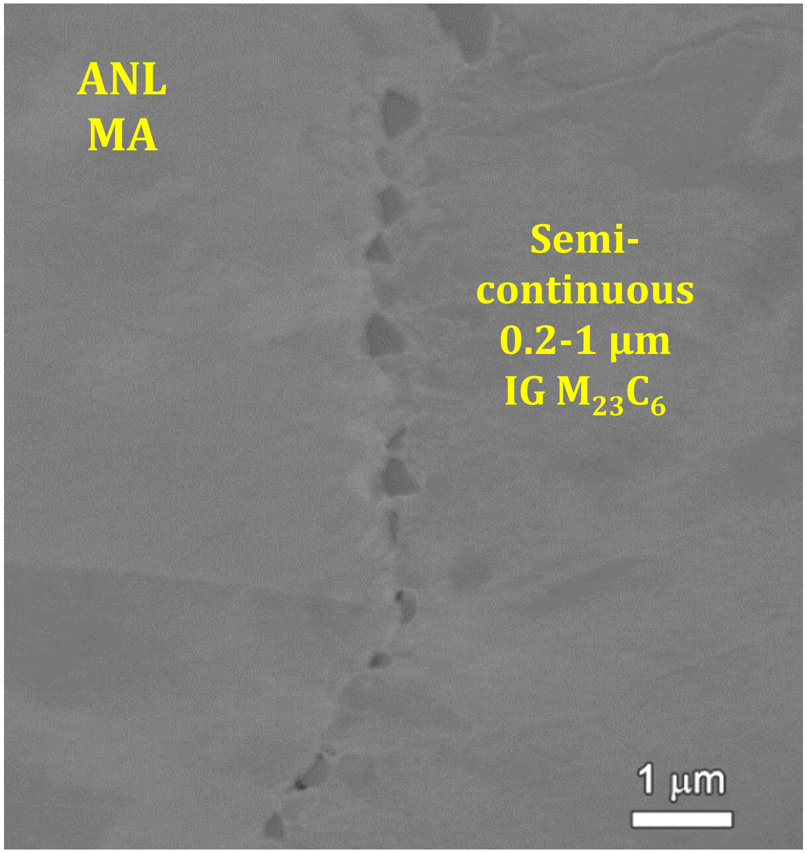


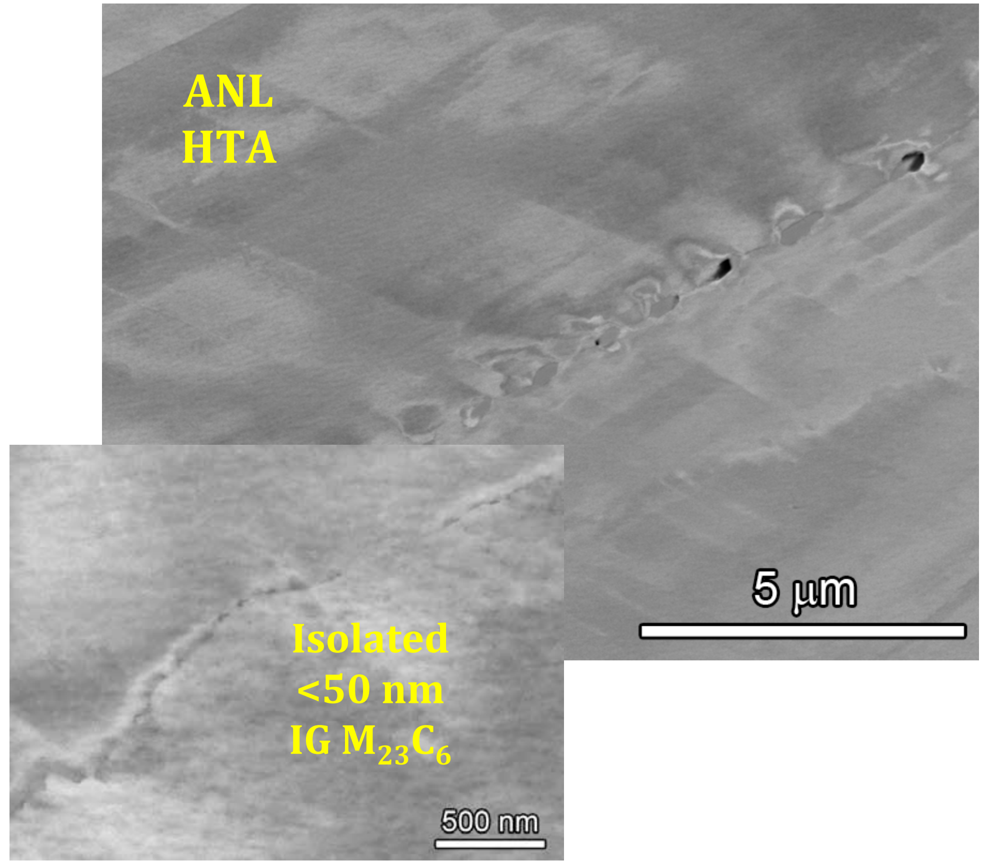
ANL HTA + 30%CF: Average Vickers Hardness = 317 kg/mm^2



ANL HTA + 30%CF plate has local regions of larger grains with lower twin density and lower average misorientation density than MA + 30%CF. Most areas show similar grain size, twinning and misorientation density plus overall average Vickers hardness is essentially identical.

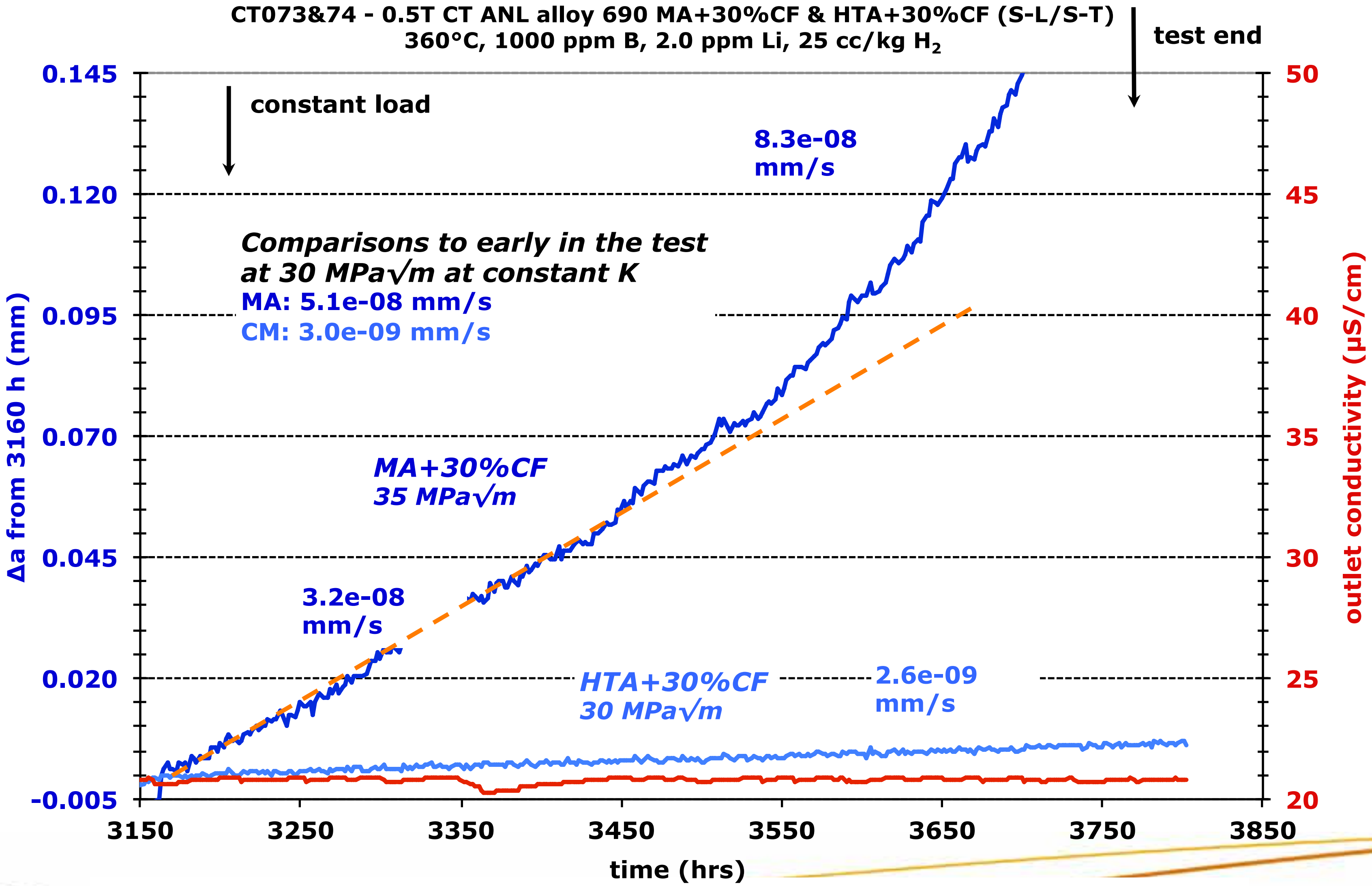
Precipitation Microstructures in ANL MA versus HTA Alloy 690 Plate





SEM (and TEM) examinations reveal that the ANL MA alloy 690 plate has a high density of µm-size Cr carbides at nearly all high-energy grain boundaries. HTA dissolves most of these carbides resulting in a much lower density of fine (5-50 nm) carbides at isolated sections of grain boundaries.

High Temperature Anneal on SCC Crack Growth for Cold-Worked Alloy 690 Plate



Initial high temperature anneal results in much lower (~15X) SCC growth rates in highly cold-worked alloy 690 plate.

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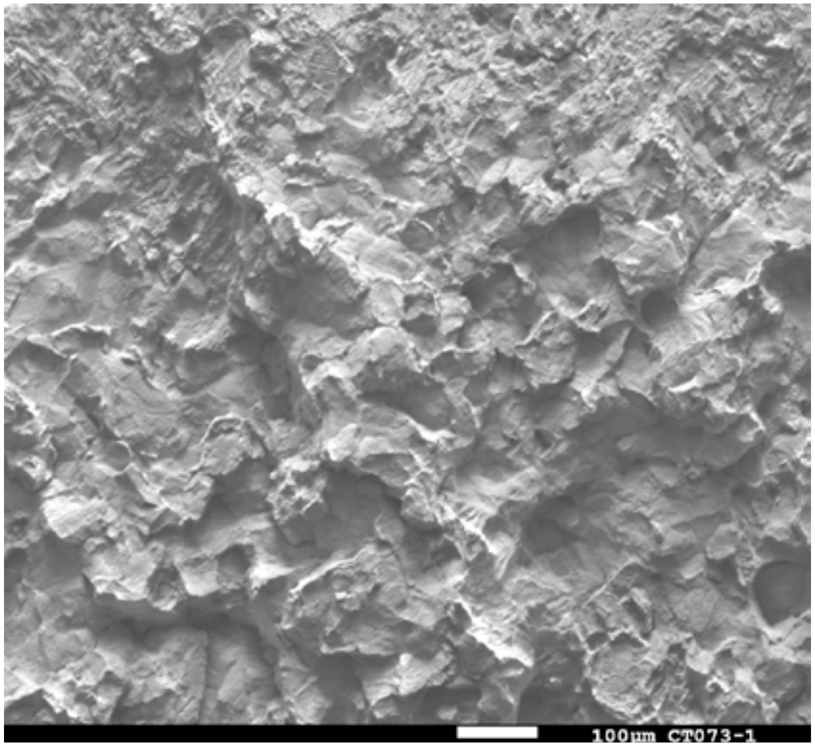
Cracking Morphology on 30%CF ANL Alloy 690: MA versus HTA Materials

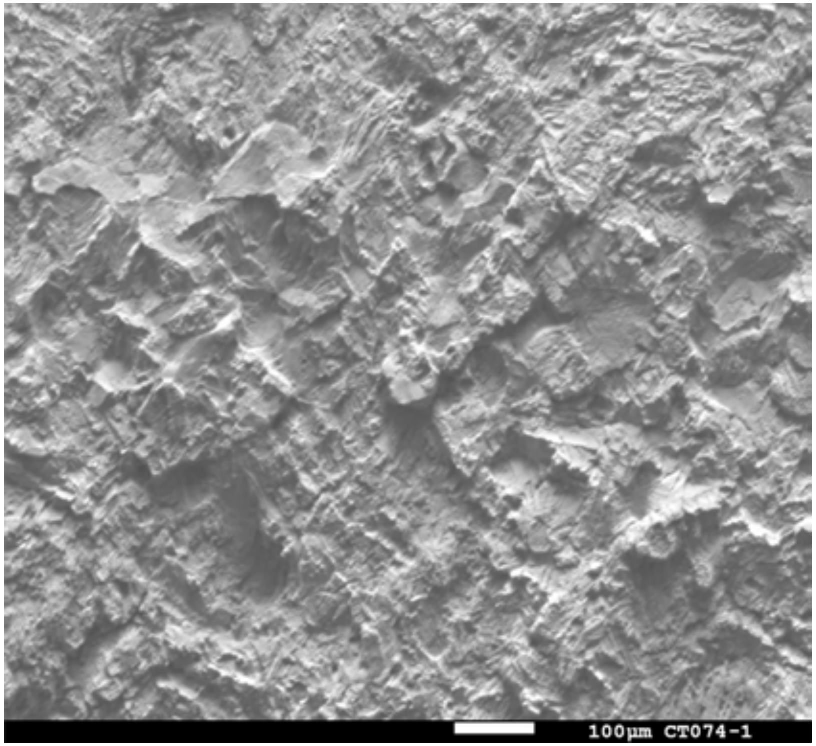
ANL MA + 30%CF

Air Fatigue

IG SCC



ANL HTA + 30%CF



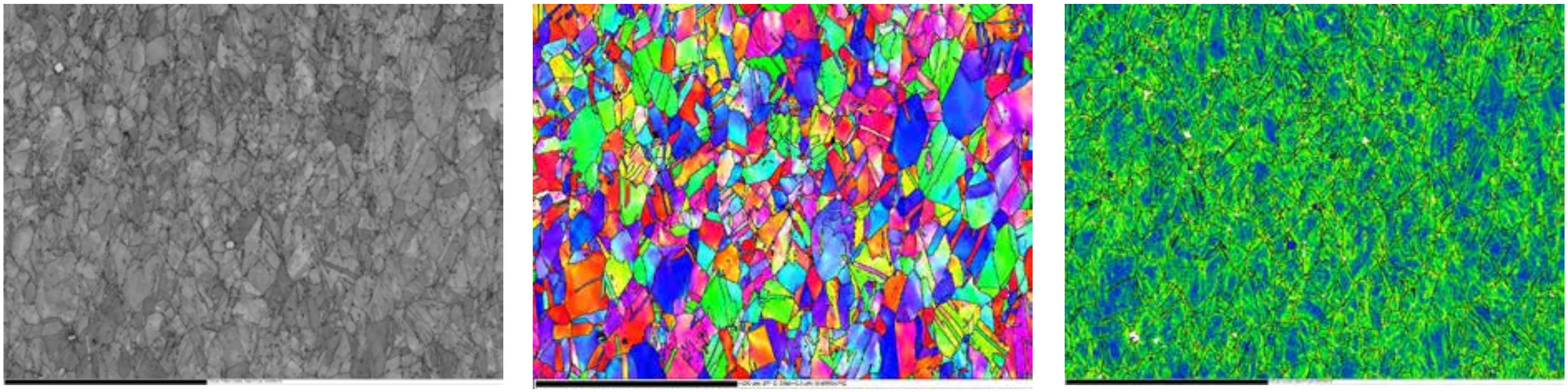
Air Fatigue

Mixed IG/TG

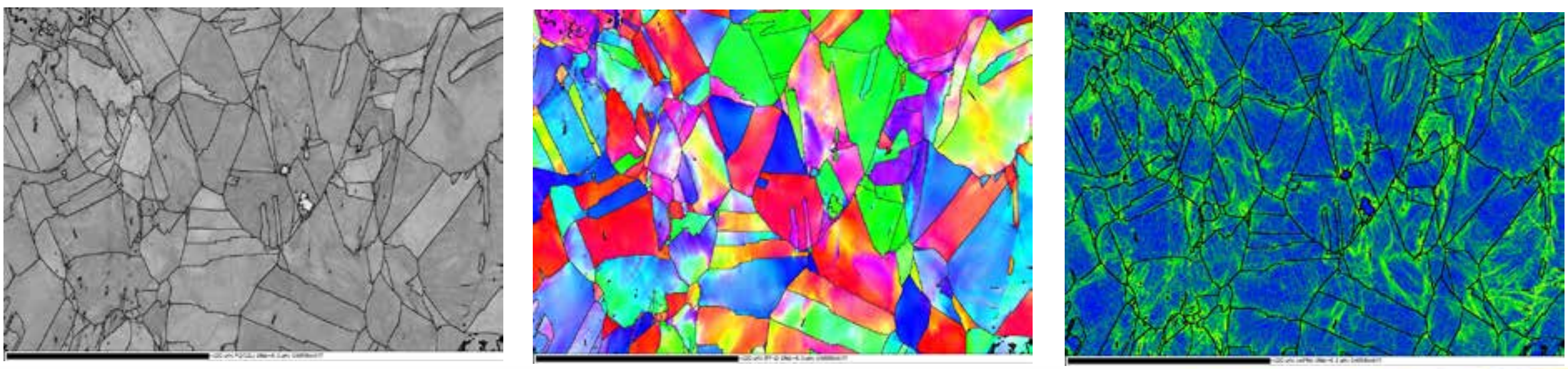
Extensive IGSCC growth is observed during the final constant K evaluation in ANL MA + 30%CF alloy 690 specimen, much more limited IG cracking is present for the ANL HTA + 30%CF alloy 690 specimen.

Microstructure and Strain Distributions in GEG MA versus HTA 20%CR Alloy 690 Plate

GEG MA + 20%CR: Average Vickers Hardness = 321 kg/mm^2



GEG HTA + 20%CR: Average Vickers Hardness = 291 kg/mm^2



GEG HTA + 20%CR plate has a larger grain size, lower twin density, lower average misorientation density and lower average hardness than GEG MA + 20%CR.

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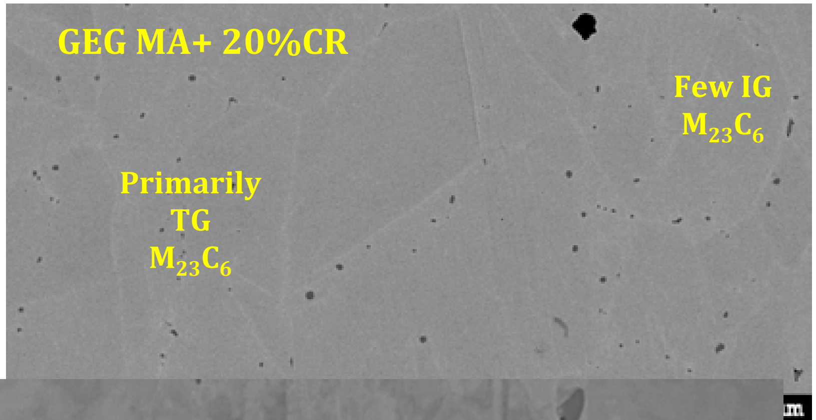
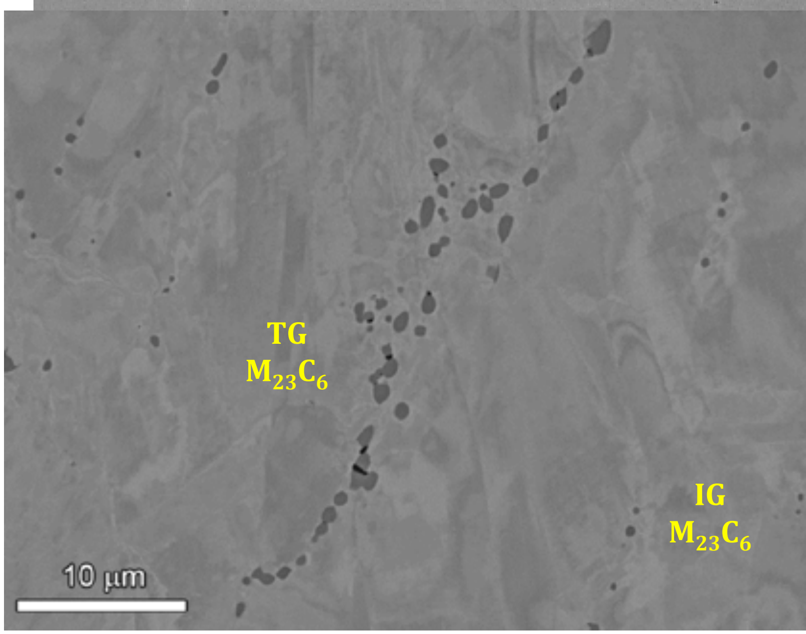
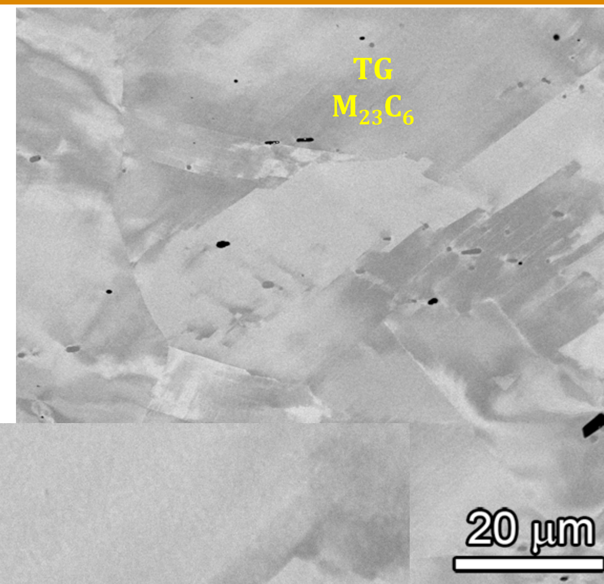
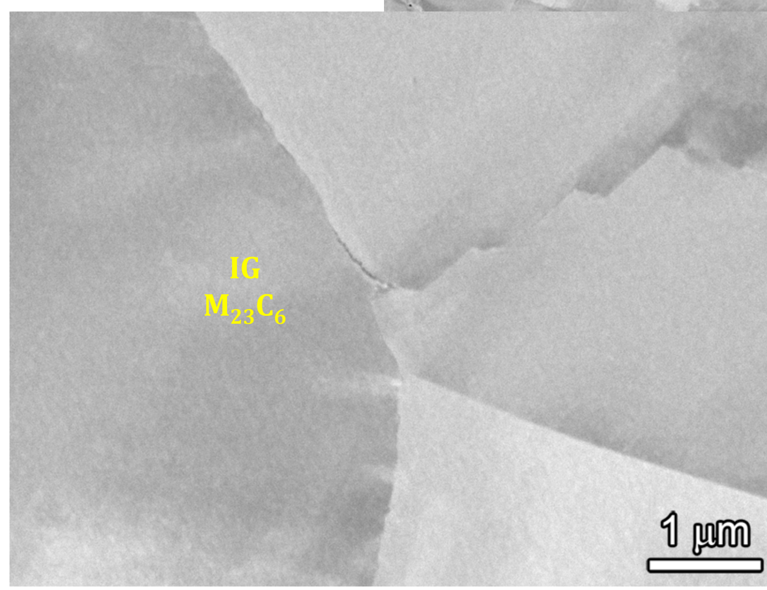
Precipitation Microstructures in GEG MA versus HTA Alloy 690 Plate





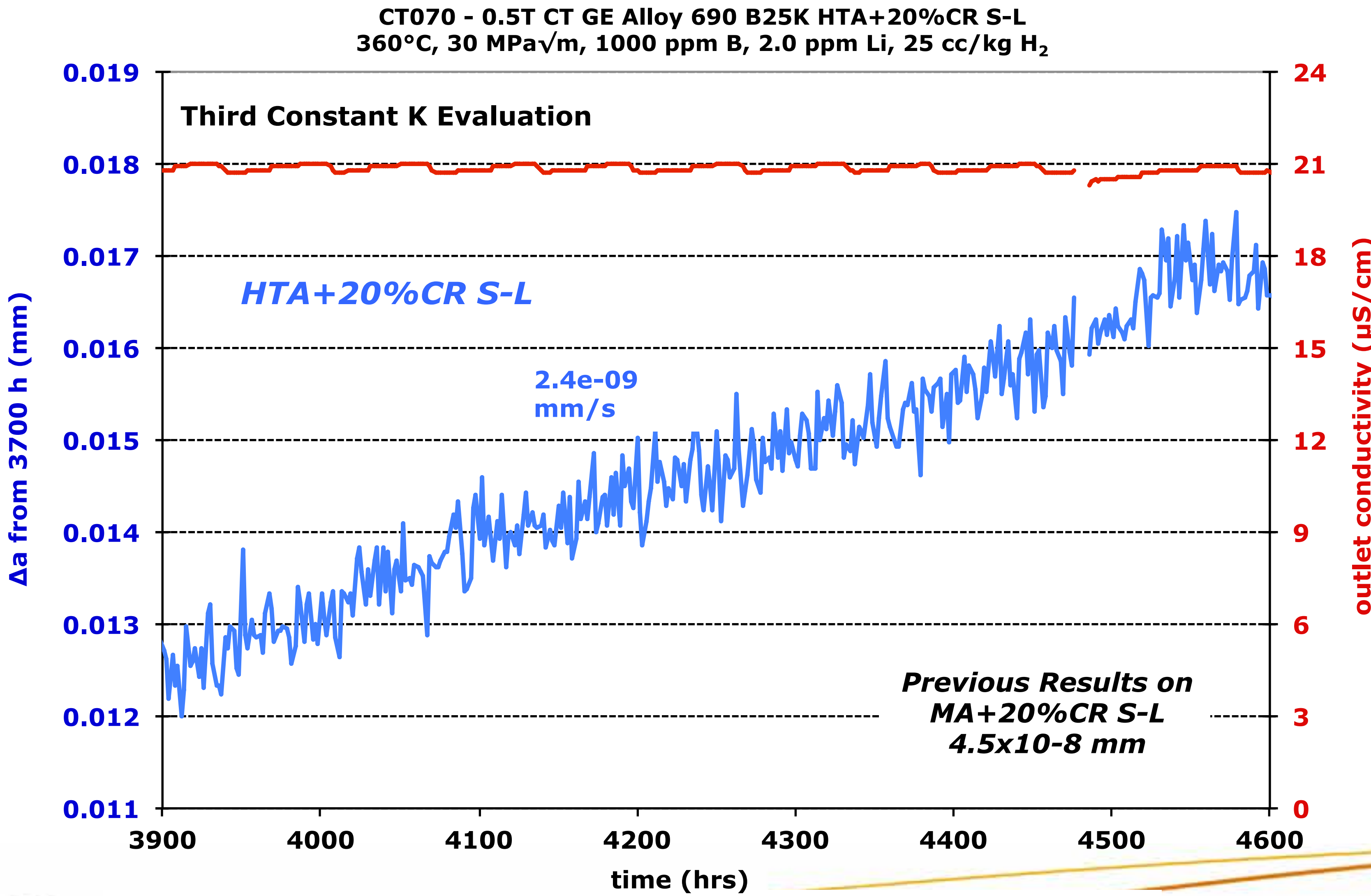
GEG HTA +
20%CR





GEG MA plate has a significant microstructural variability, finer grain size, matrix carbides and a low density of carbides on ***most*** *grain boundaries. HTA alters microstructure and dissolves carbides, results in a low density of fine (5-50 nm) carbides at isolated sections of grain boundaries.*

High Temperature Anneal on SCC Crack Growth for Cold-Worked Alloy 690 Plate



Initial high temperature anneal results in much lower (~20X) SCC growth rates in 20%CR alloy 690 plate.

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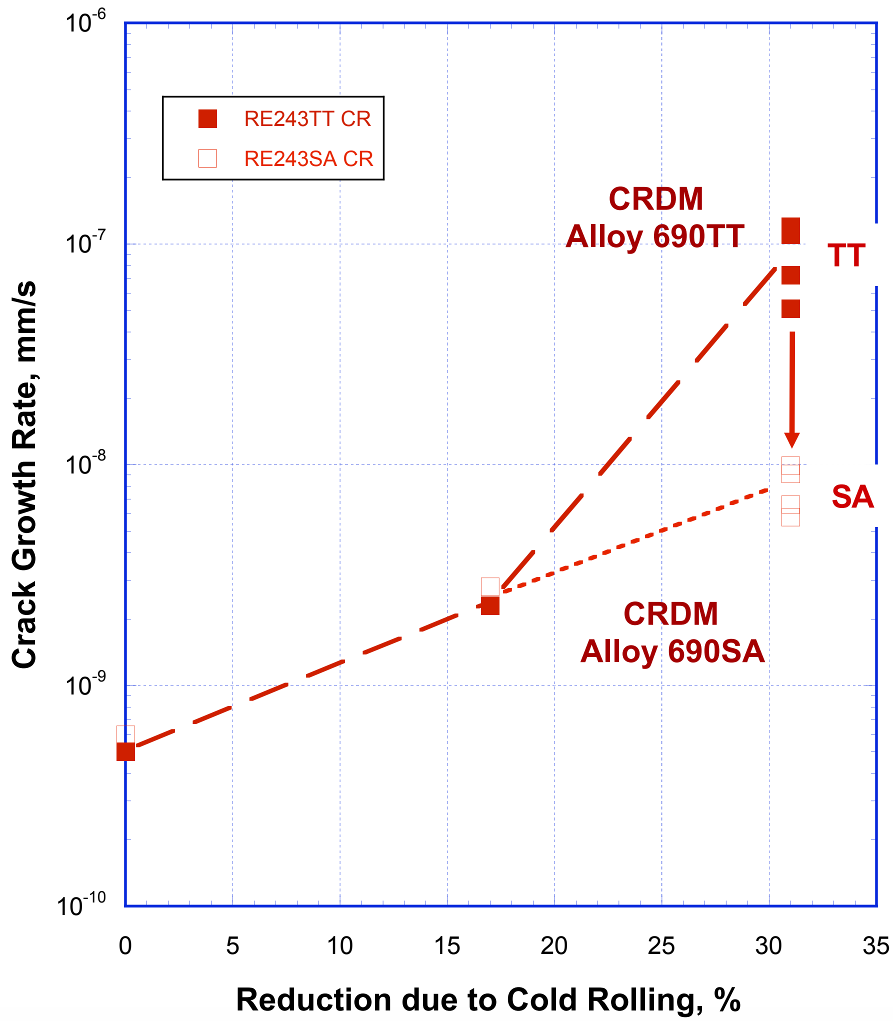
Influence of Initial High Temperature Anneal on Alloy 690 Microstructure and SCC

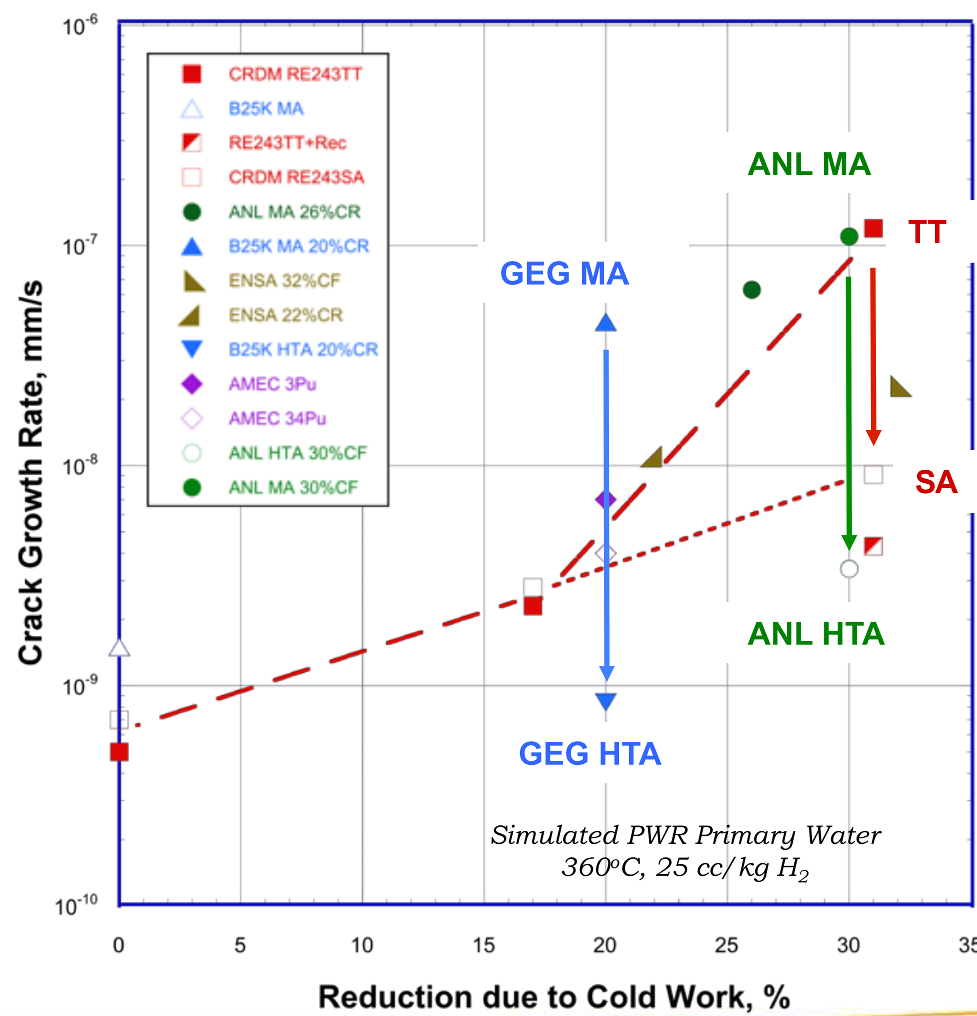
Alloy 690 Material	Microstructure	Test Plane Hardness	PNNL CGRs
TT+31%CR	Nearly continuous GB carbides, isolated GB and matrix TiN, elongated grains, very high dislocation density, moderate density of IG voids, some cracked carbides and TiN	315 kg/mm^2	$1.2x10^{-7}$ mm/s (S-L, 360°C)
SA+31%CR	Isolated GB and matrix TiN, elongated grains, very high dislocation density, few cracked TiN	316 kg/mm^2	$9.7x10^{-9}$ mm/s (S-L, 360°C)
ANL Bar MA+30%CF	Semi-continuous GB carbides and TiN, matrix TiN, slightly elongated grains, high dislocation density, moderate-to-high density of IG cracked carbides/TiN precipitates and voids	316 kg/mm^2	$8.2x10^{-8}$ mm/s (S-L, 360°C)
ANL Bar HTA+30%CF	Fine GB carbides and TiN, matrix TiN, slightly elongated grains, high dislocation density, low density of IG voids, cracked TiN	317 kg/mm^2	$3.0x10^{-9}$ mm/s (S-L, 360°C)
GEG Billet B25K MA+20%CR	Occasional GB carbides and TiN with more in banded regions, many matrix carbides and TiN, slightly elongated grains, high dislocation density, isolated IG voids, cracked carbides	321 kg/mm^2	$4.5x10^{-8}$ mm/s (S-L, 360°C)
GEG Billet B25K HTA+20%CR	Fine GB carbides and TiN, slightly elongated and larger grains, high dislocation density, isolated IG voids, cracked carbides	291 kg/mm^2	$2.4x10^{-9}$ mm/s (S-L, 360°C)

Preliminary Data

Initial HTA treatment produces a consistent decrease in SCC growth response for highly cold-worked alloy 690 materials.

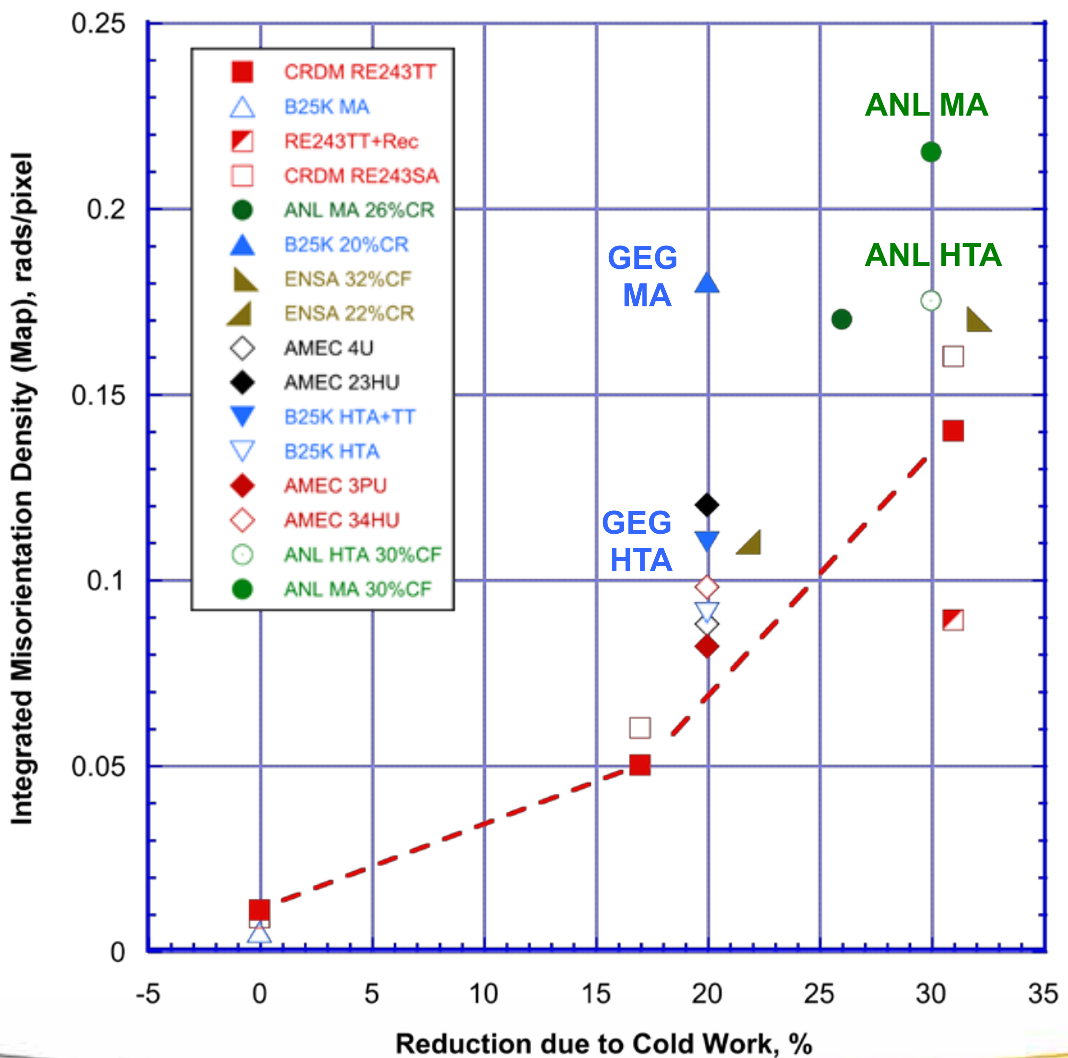
SCC Crack Growth Rates for Cold-Worked Alloy 690 CRDM and Plate Materials





SCC growth rate does not scale with %CW, different behavior indicated at high levels of cold work for CRDM and plate materials with important effect of initial material condition.

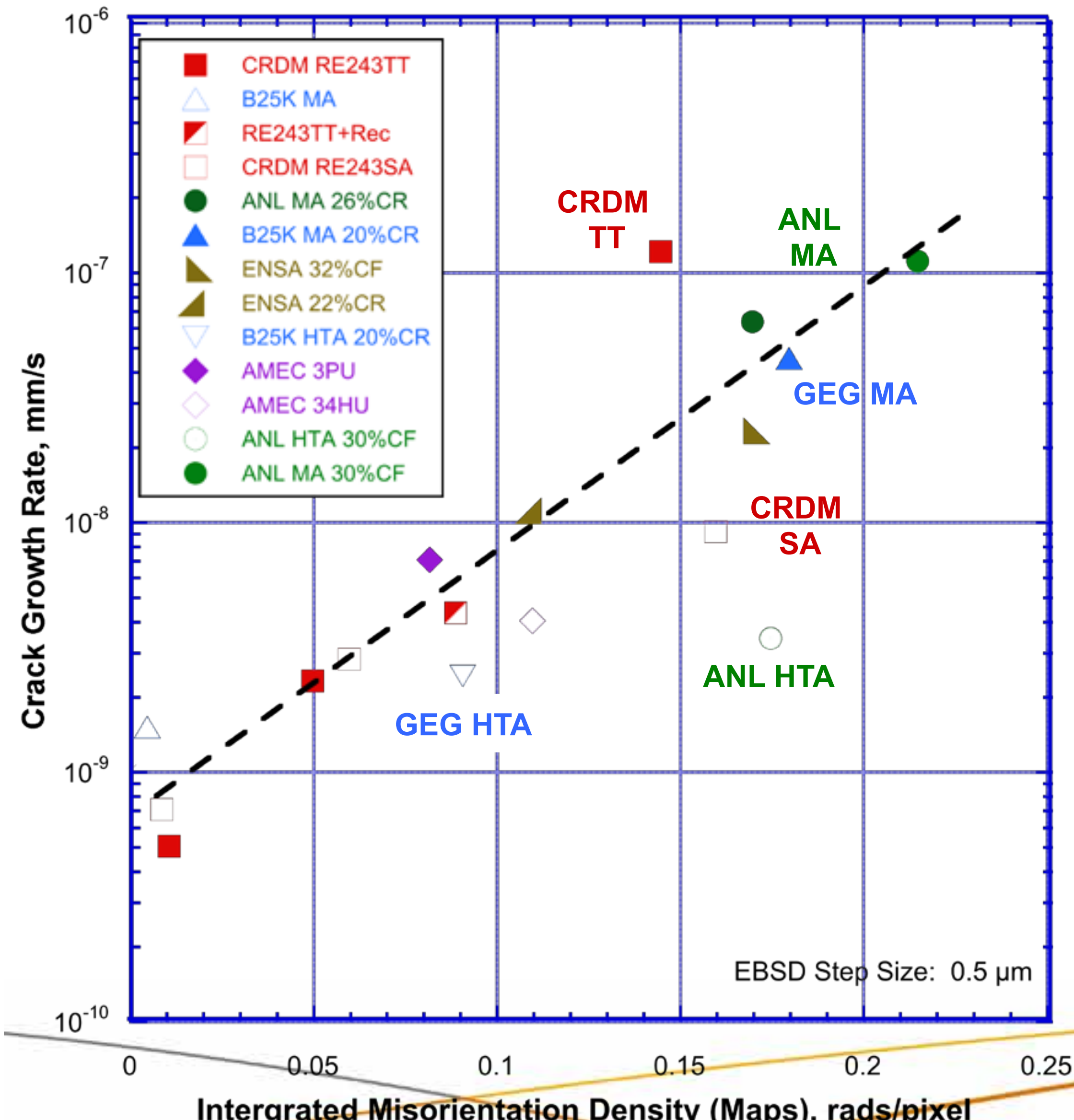
EBSD Measurements of Average Misorientation in CW Alloy 690 Materials



Reasonably good correlation for most materials using average EBSD misorientation densities except for cold-worked GEG and ANL MA plate materials. Cold-worked GEG and ANL plates fit general correlation if given initial HTA treatment.

Clearly initial material condition and microstructure can influence subsequent strain distribution from cold working.

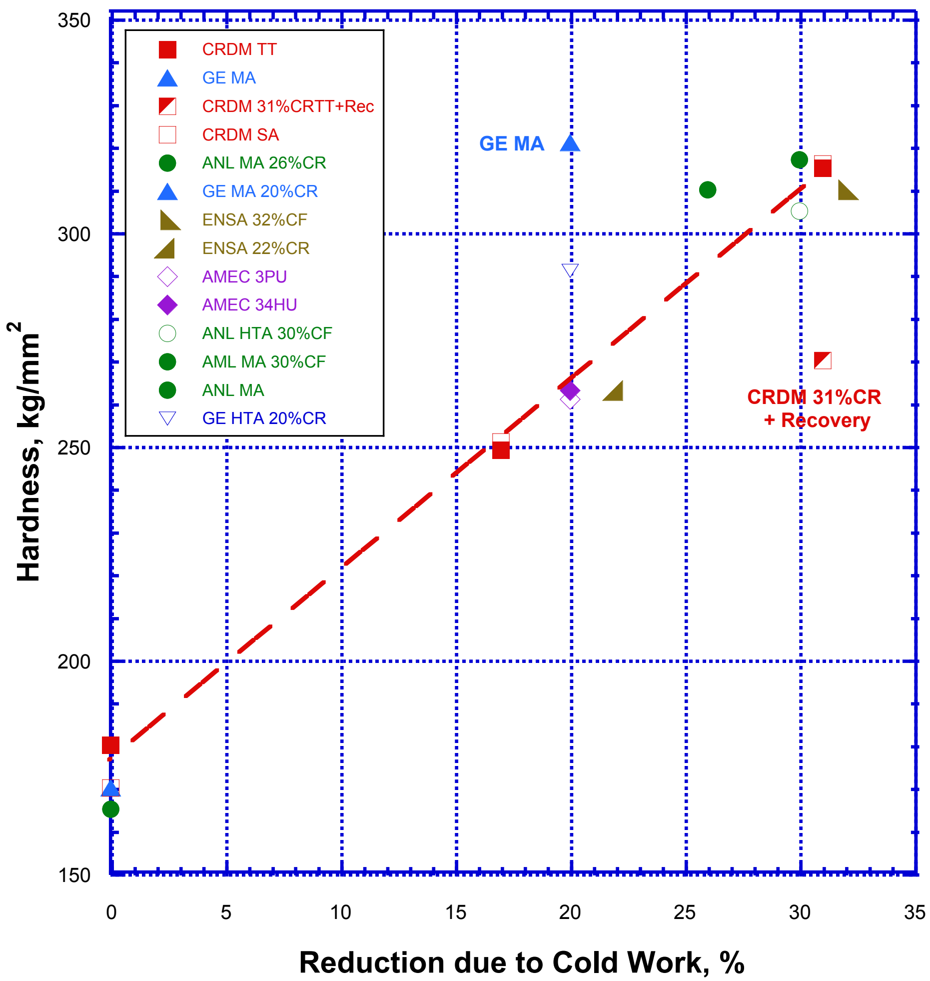
Correlations Between Average EBSD Misorientation Density and SCC Growth Rates

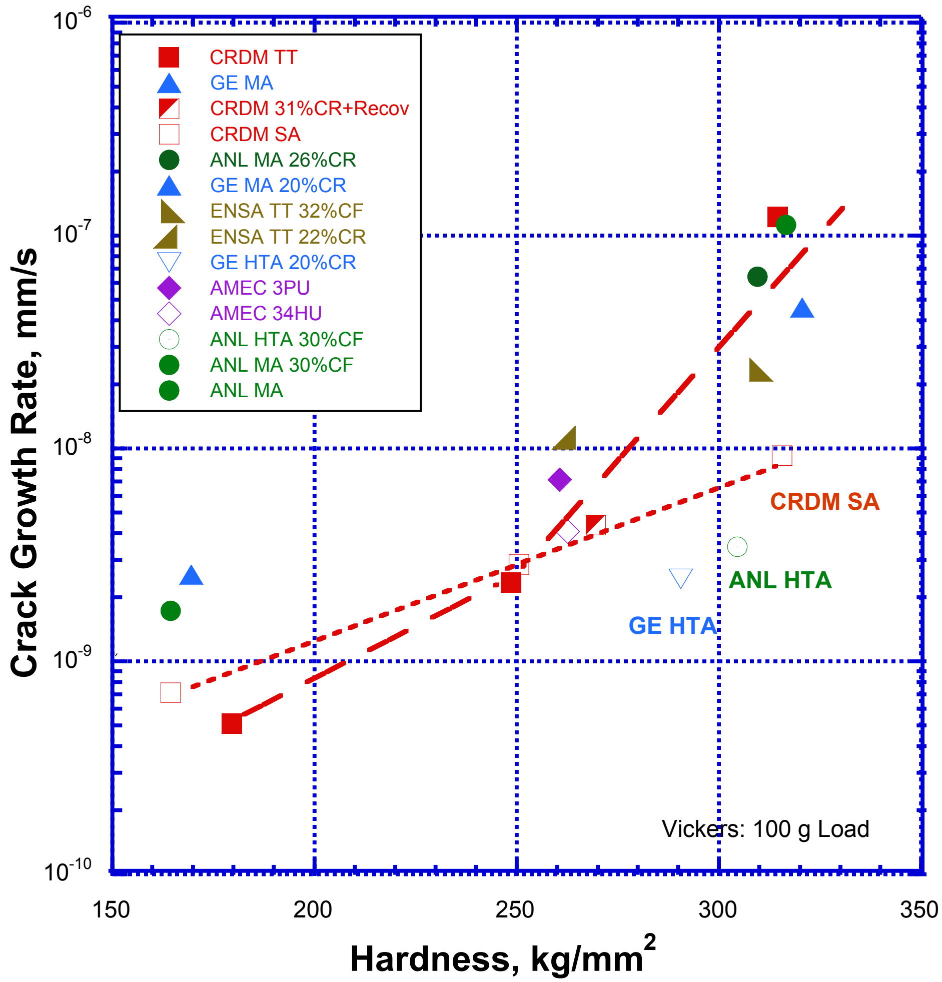


Good correlation with SCC growth rates for most cold-worked alloy 690 heats including 20%CR GEG, 26%CR ANL, 30%CF ANL and 31%CR CRDM + recovery.

However, HTA + CW materials show lower SCC growth rates at equivalent EBSD strains.

Correlations Between Hardness and SCC Growth Rates





SCC correlation with hardness also helps explain outlier points for 31%CR + Recovery and 20%CR GEG specimens. HTA + CW materials again show lower SCC growth rates at equivalent hardness values.

Microstructure Effects on IGSCC in Cold-Worked Alloy 690 Materials

- *Results demonstrate that alloy 690 tubing and plate materials with different starting microstructures become susceptible to IGSCC in the highly deformed ($\geq$~20%CR) condition. Significant heat-to-heat and material condition differences in SCC susceptibility are observed for these highly CW materials.*
- *High levels of CW produces slightly elongated grains, high dislocation densities particularly at grain boundaries and some degree of IG void formation and cracked carbides/nitrides depending on the precipitate distribution. Pre-existing grain boundary voids and cracks do not directly promote IGSCC.*
- *Best correlation to SCC growth rates is for EBSD-measured strains and hardness in the alloy 690 materials. Current data indicates that matrix strength and deformation structures near grain boundaries control IGSCC susceptibility.*
- *Initial high temperature anneal and quench improves SCC resistance in highly CW alloy 690 heats. It appears that this results from the removal of pre-existing matrix damage and/or modification of the grain boundary carbide distribution.*