Cold Work Effects on Stress Corrosion Crack Growth in Alloy 690 Tubing and Plate Materials

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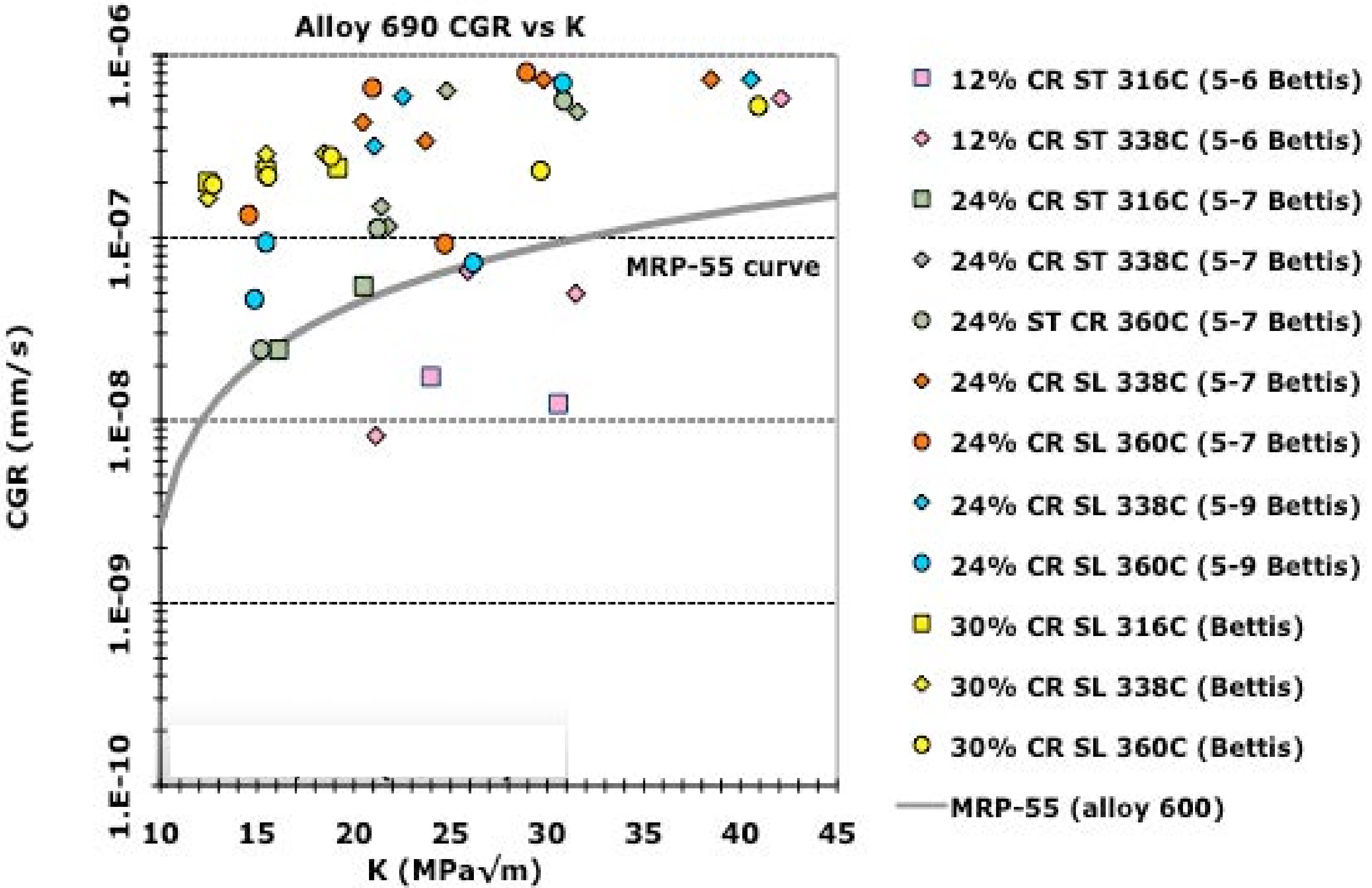
Outline

- *Review of Laboratory Alloy 690 Stress Corrosion Growth Rate Data in Simulated PWR Primary Water*
- *PNNL Investigation of Cold Work Effects on Alloy 690 Stress Corrosion Growth Rates*
  - *As-received CRDM, plate and bar materials*
  - *Low levels of cold work: 11-17%*
  - *Moderate levels of cold work: 20-22%*
  - *High levels of cold work: 26-32%*
- *Correlations Among Hardness, Strain Distributions and Stress Corrosion Growth Rates*
- *Effects of Initial Microstructure and Warm Work*
- *Summary and Conclusions*

SCC Susceptibility of Cold-Worked Alloy 690 in PWR Primary Water



Potential concern for cold-work effects on alloy 690 SCC first identified by Bettis in presentations ~10 years ago.

SCC Susceptibility of Cold-Worked Alloy 690 in PWR Primary Water

- *Concern for cold-worked (CW) alloy 690 identified by Bettis, reported in presentations ~10 years ago*
- *Multi-laboratory investigations have confirmed IGSCC susceptibility in <u>highly</u> CW materials*
  - *Moderate-to-high SCC propagation rates when tested in plane of deformation (S-L/S-T orientation)*
  - *Suggestions that material compositional/microstructural inhomogeneity and 1D rolling are responsible for higher SCC susceptibility have not been established.*
  - *In general, similar response for extruded CRDM and plate heats in highly CW condition however data limited for low CW levels possibly relevant to PWR service.*
- *PNNL research has focused on 12 well-characterized alloy 690 CRDM, bar and plate heats as a function of cold work including as received, low (11-17%), moderate (18-24%) and high (25-32%) levels.*

PNNL Alloy 690 Testing Summary

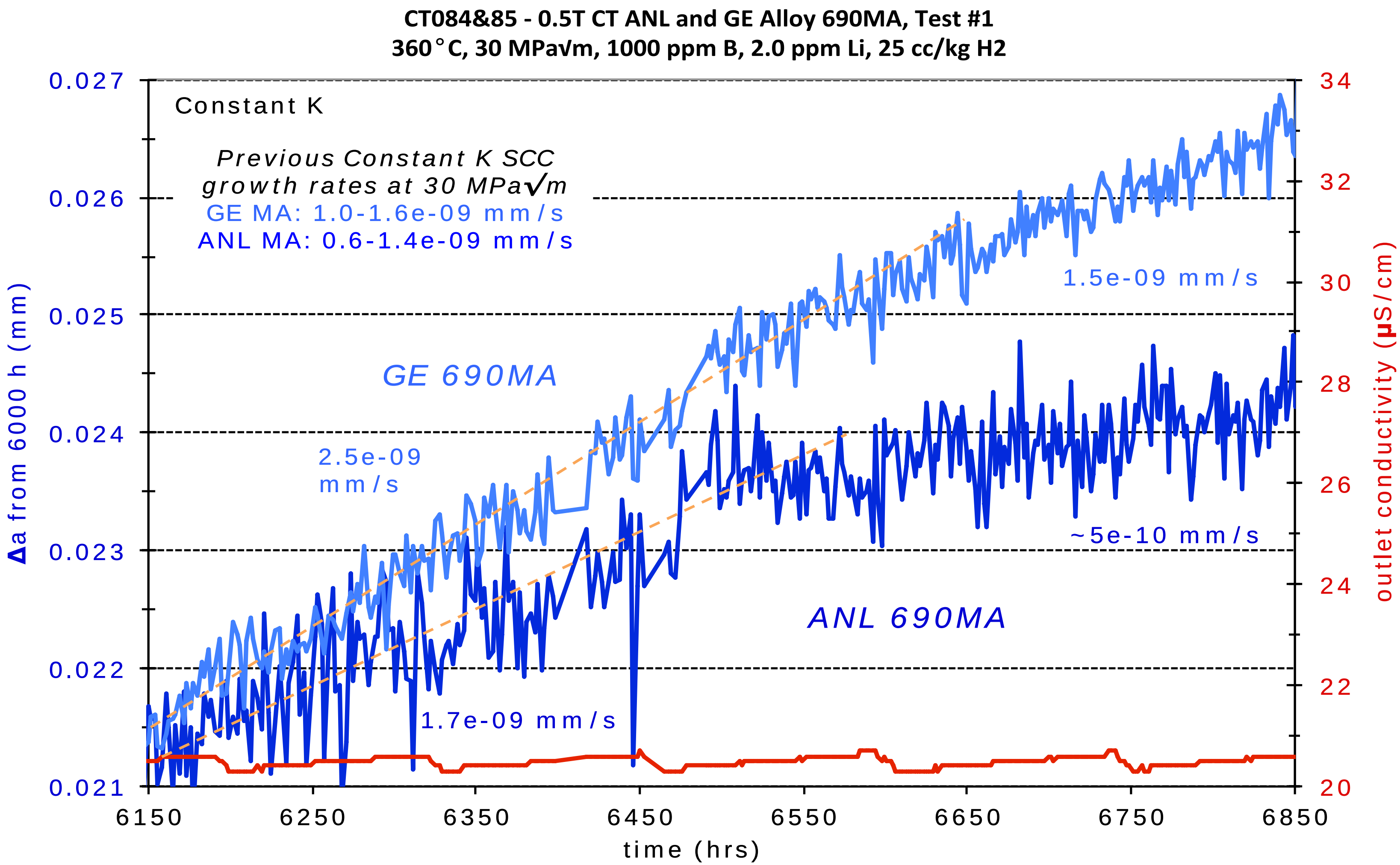
Alloy 690 CRDM (21 tests)

- *As-received TT heats: Valinox RE243, WP140 and WP142; Doosan 133454; Sumitomo E67074C*
- *As-received TT (high density of IG Cr carbides, Cr depletion) versus HTA/SA for Valinox RE243 + 0%, 17%CR, 30%CR, 31%CR; post-CW recovery anneal: 31%CR alloy 690TT + 700°C/1h*
- *As-received TT + 12%CF, 21%CF & 30%CF Valinox RE243; 20%TS Valinox (WP787); 13%CF & 30%CF Sumitomo; 21%CF Doosan*

Alloy 690 Plate/Bar Materials (13 tests)

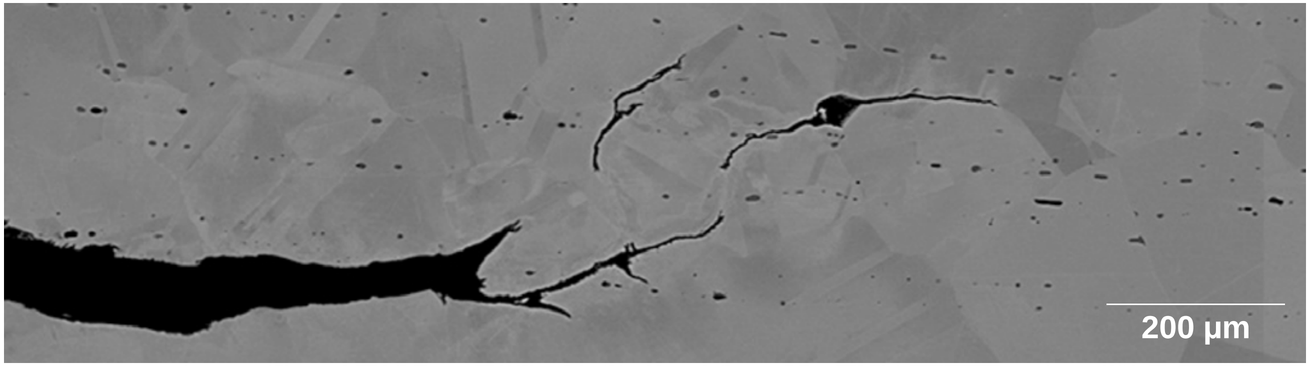
- *As-received MA heats: ANL (NX3297HK12), GEG (B25K), TK-VDM (114092), ENSA Divider Plate (TT)*
- *MA versus HTA for 30%CF ANL and 20%CR GEG heats*
- *As-received + 21%CF Allvac (X87N-1), 22%CR & 30%CF ENSA (SP547), 26%CR ANL, 32%CF TK-VDM*

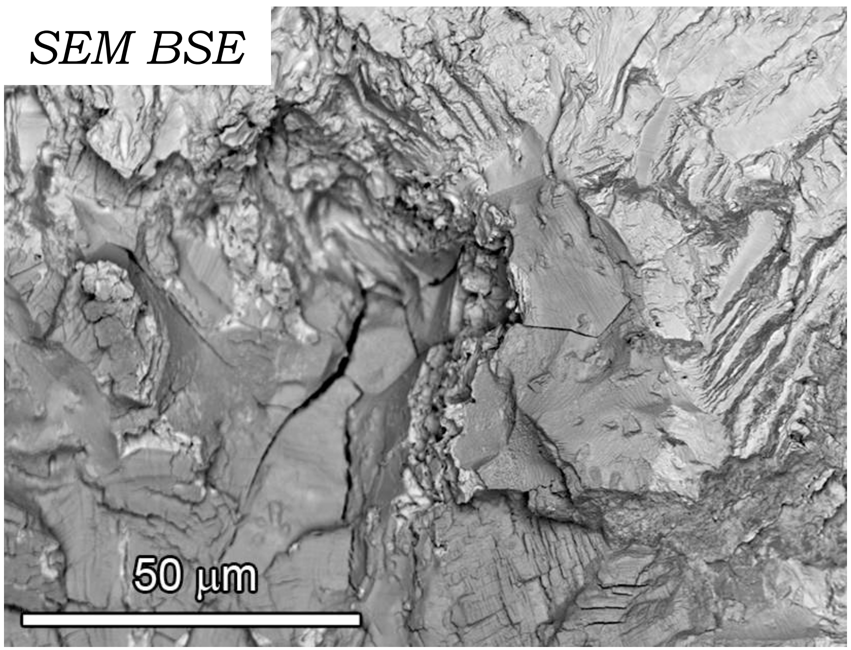
SCC Crack Growth Response for Non-CW MA Alloy 690 Plate and Bar Heats

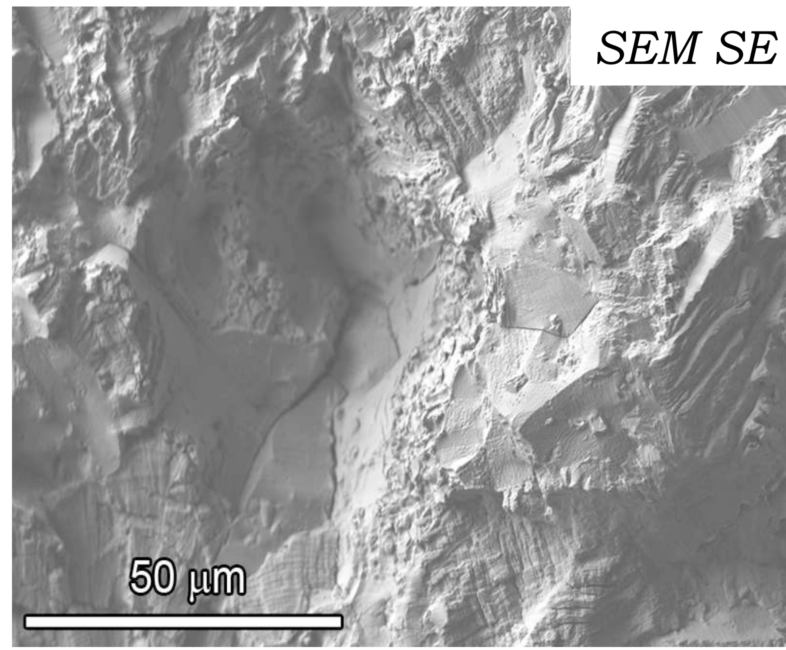


Extremely low crack-growth rates during constant K evaluations, slightly higher for GEG versus ANL MA heat. Typical response showing decreasing growth rate with time. No evidence for ligament/contact formation.

Evidence for IGSCC in Non-CW, MA GEG Alloy 690 Heat B25K

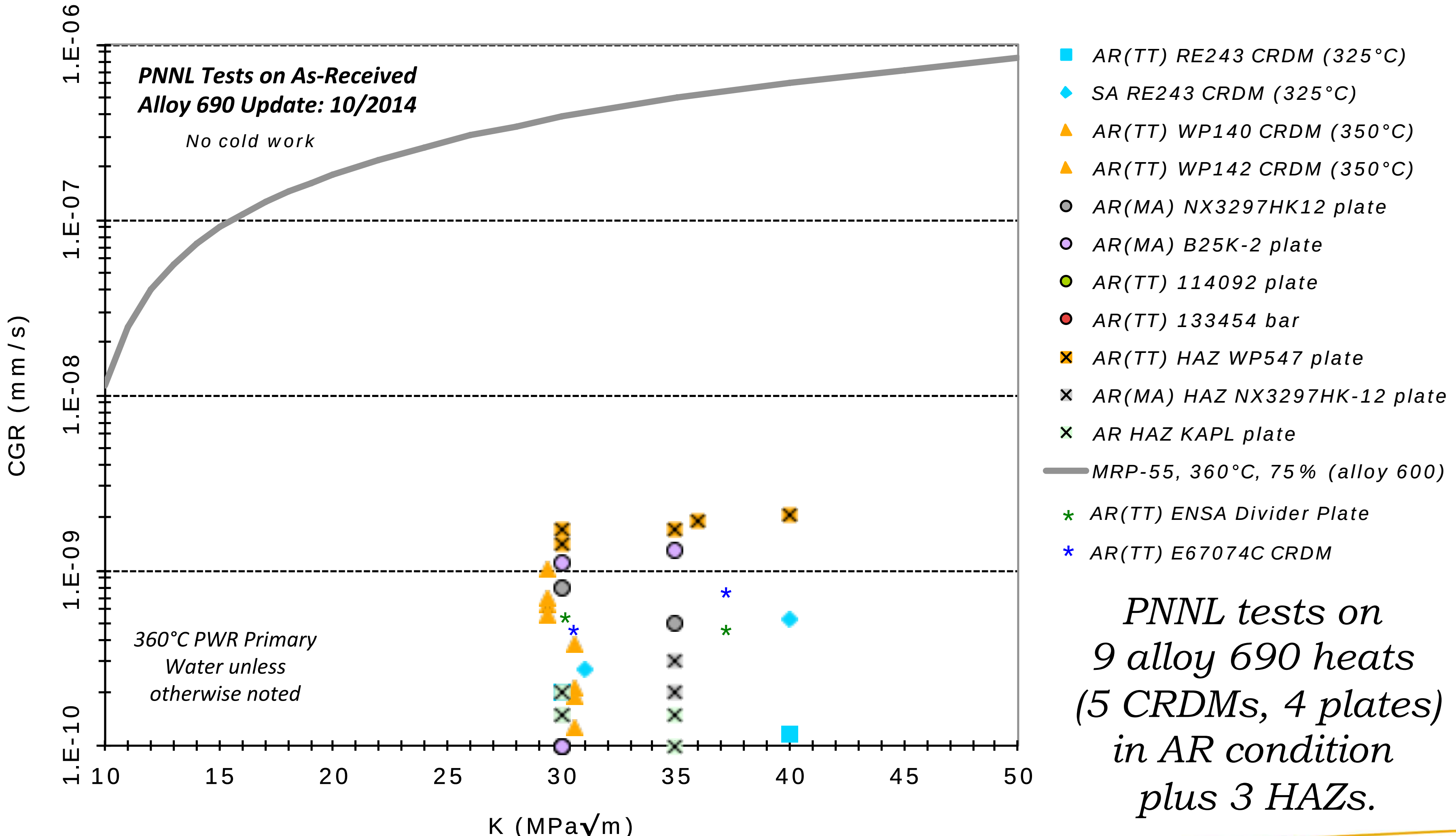






Significant regions of IG cracking were observed on the crack growth surface of the GEG specimen, overall IG engagement during final cycle + hold and constant K estimated at ~30%.

PNNL Measured SCC Growth Rates for Alloy 690 Materials without Cold Working



PNNL tests on 9 alloy 690 heats (5 CRDMs, 4 plates) in AR condition plus 3 HAZs.

Measured SCC growth rates are low or very low on alloy 690 materials in the non cold-worked condition, no significant enhanced SCC susceptibility for HAZ regions.

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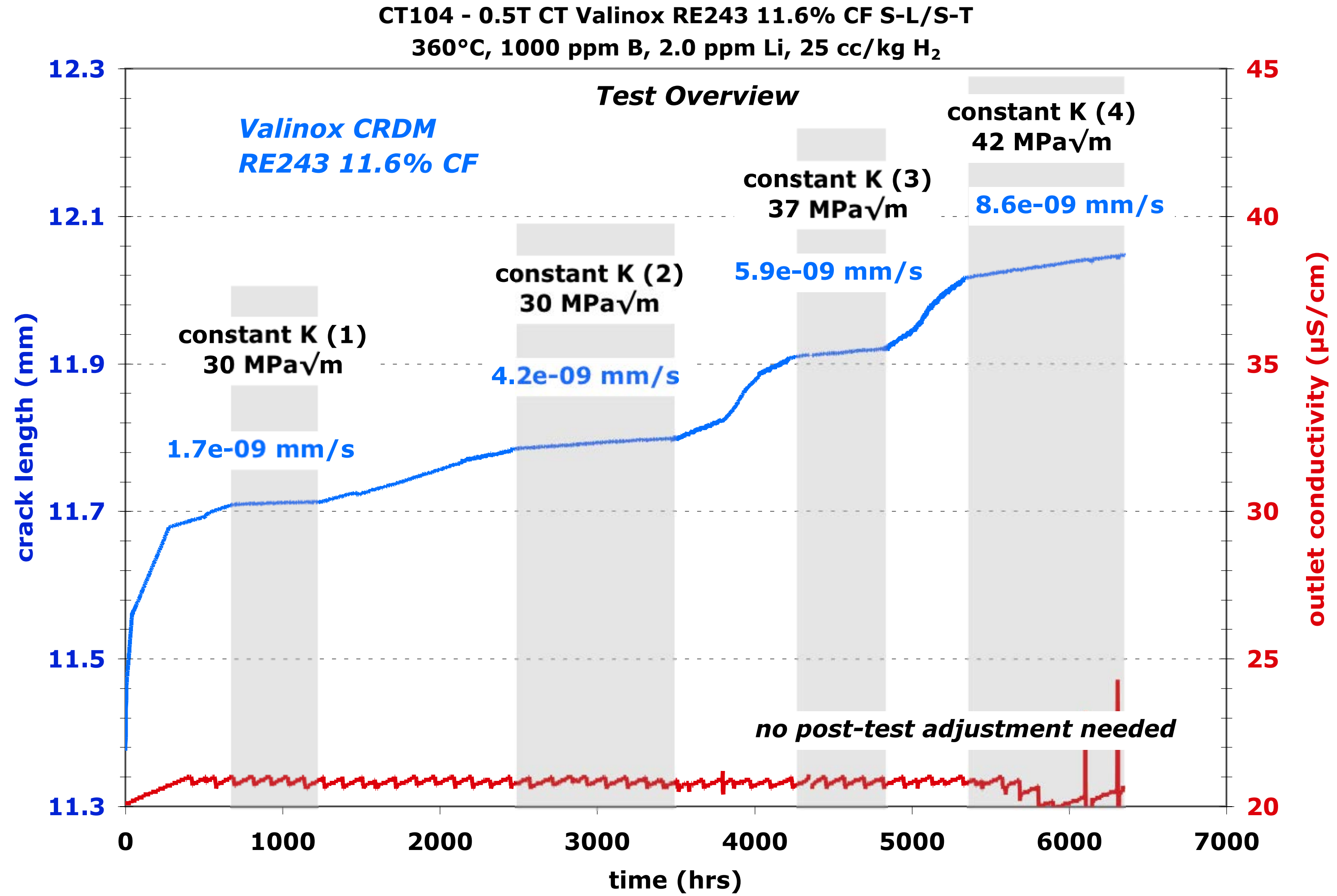
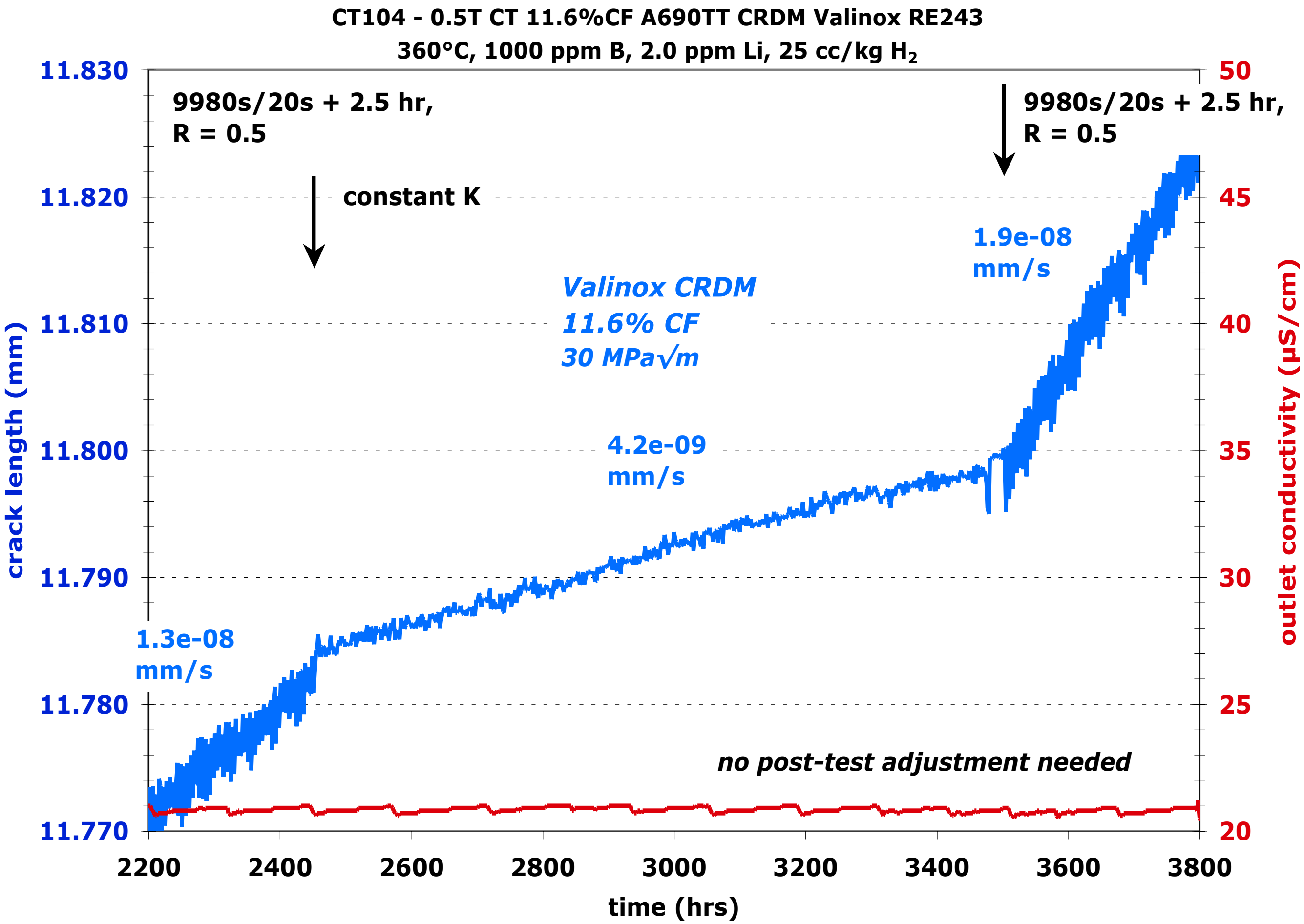
SCC Crack Growth Response for the 11.6%CW Alloy 690TT CRDM Material

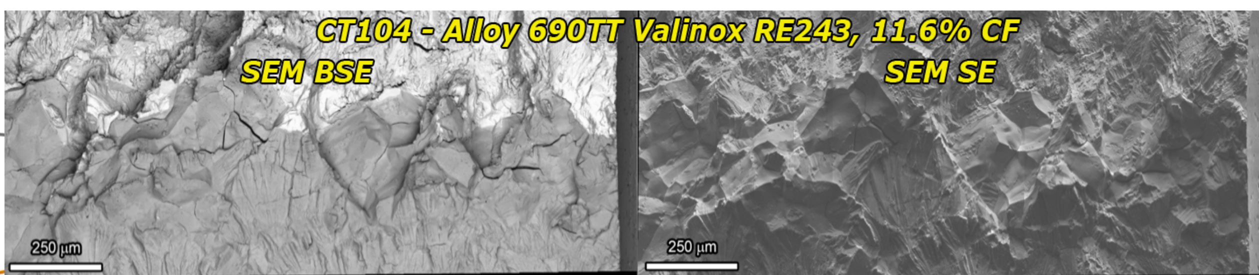


Low crack-growth rates during multiple constant K evaluations, however improved IG engagement and higher SCC rates at higher K levels.

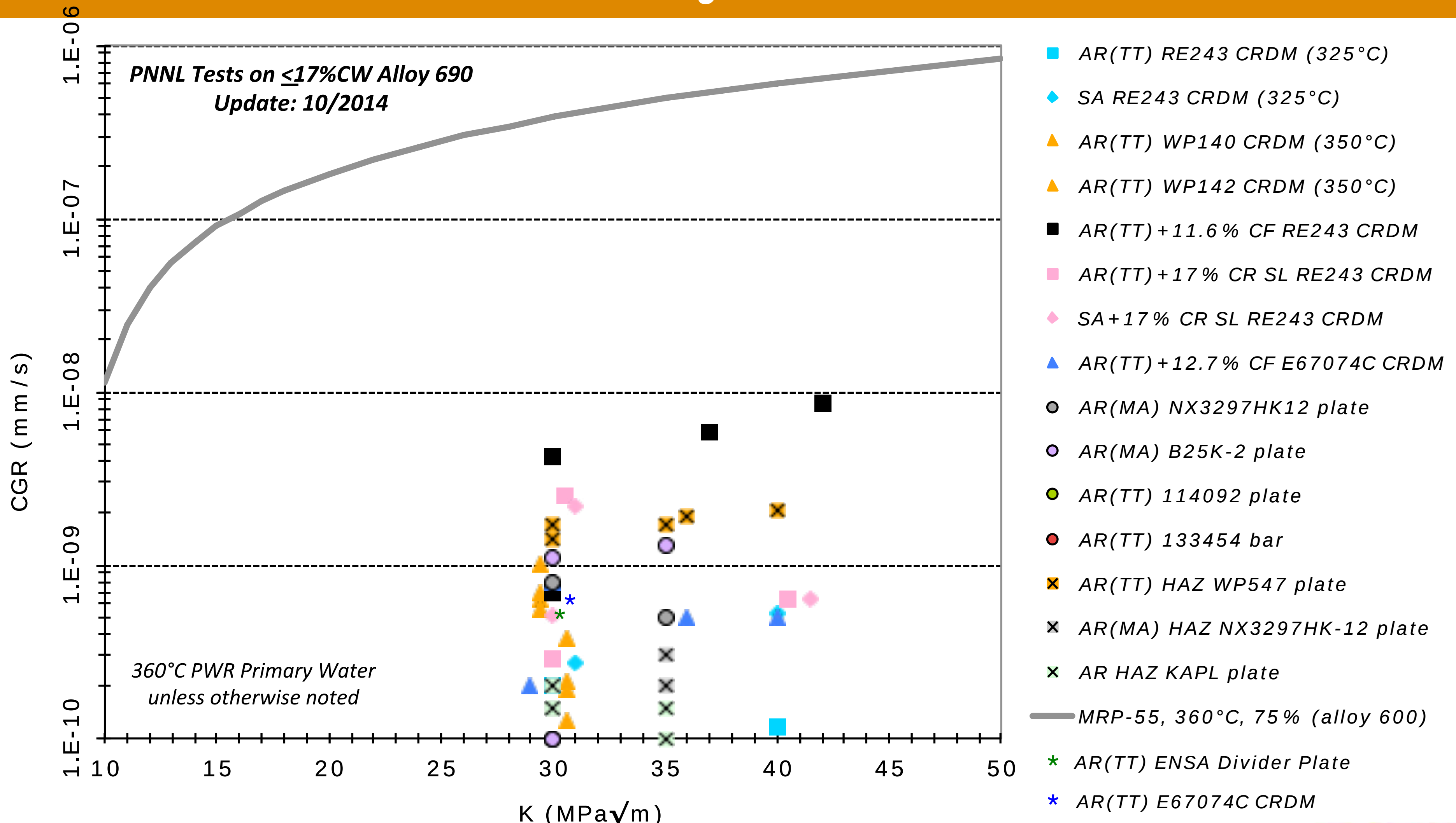
SCC Crack Growth Response for the 11.6%CW Alloy 690TT CRDM Material



Stable SCC growth observed during multiple constant K evaluations with rates 2-4x10^{-9} mm/s at moderate K. IG engagement reaches ***~80%*** *at higher K with no evidence for ligament formation.*

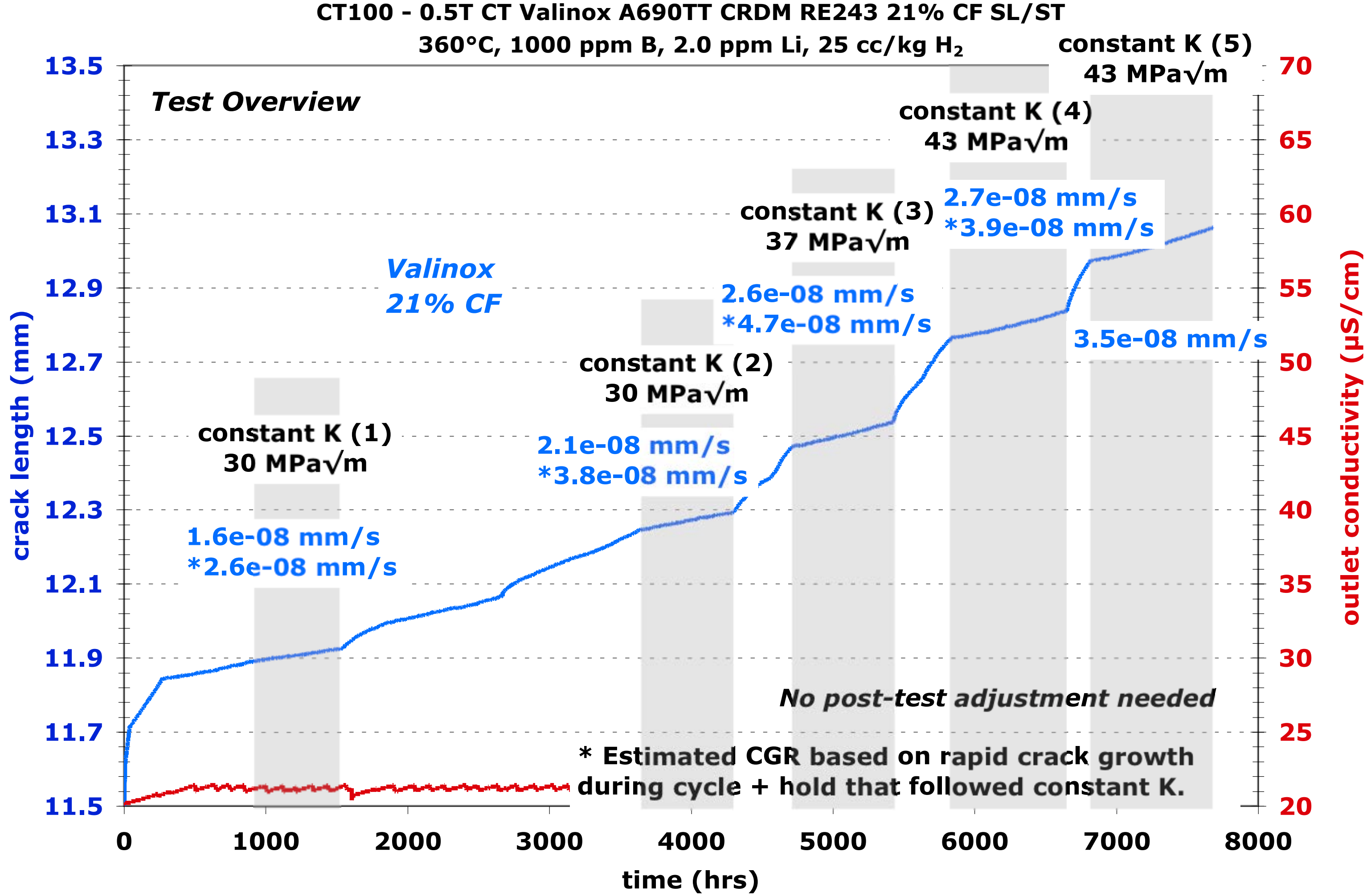


PNNL Measured SCC Growth Rates for 0-17%CW Alloy 690 Materials



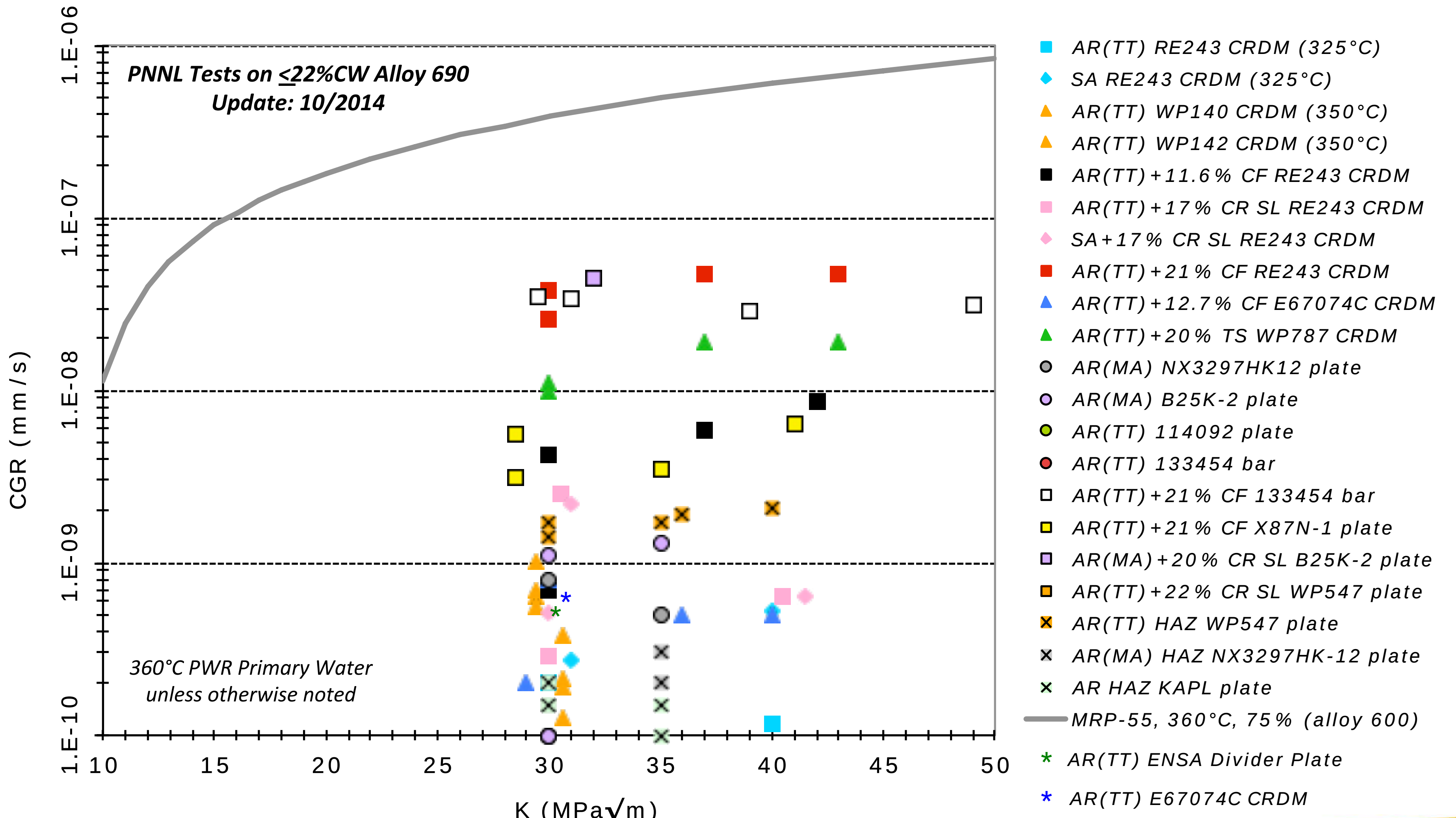
Moderate levels of cold work (11-17%) can increase SCC susceptibility in CRDM and plate heats, however propagation rates remain below ~1x10^{-8} mm/s.

SCC Crack Growth Response for the 21%CW Alloy 690TT CRDM Material



Moderate crack-growth rates during constant K evaluations for 21%CW CRDM heat with ~85% IG engagement. SCC growth rates adjusted to account for ligament/contact formation.

PNNL Measured SCC Growth Rates for 0-22%CW Alloy 690 Materials



Moderate levels of cold work (~20%) can significantly increase SCC susceptibility in CRDM and plate heats reaching propagation rates of ~5x10^{-8} mm/s.

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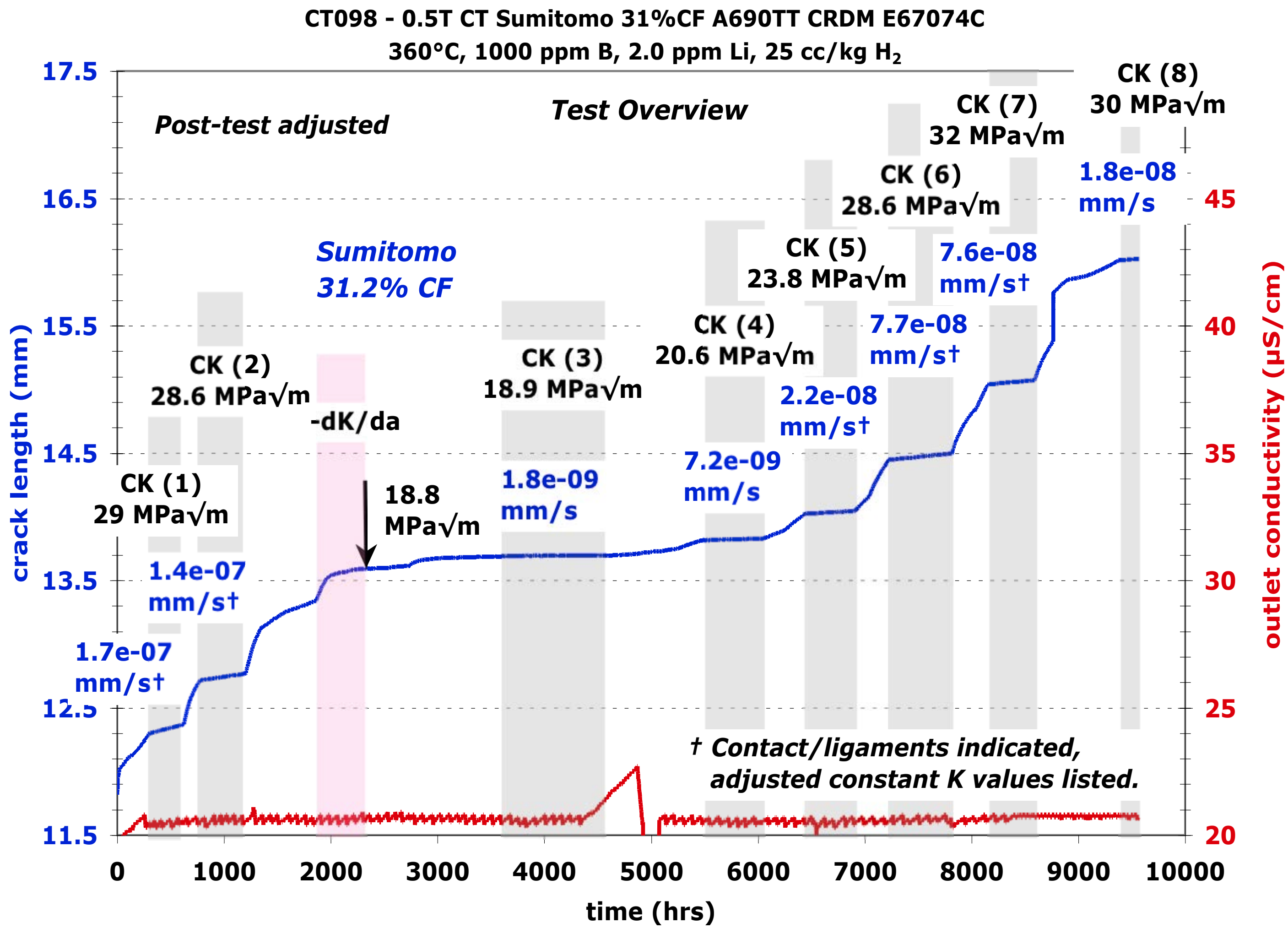
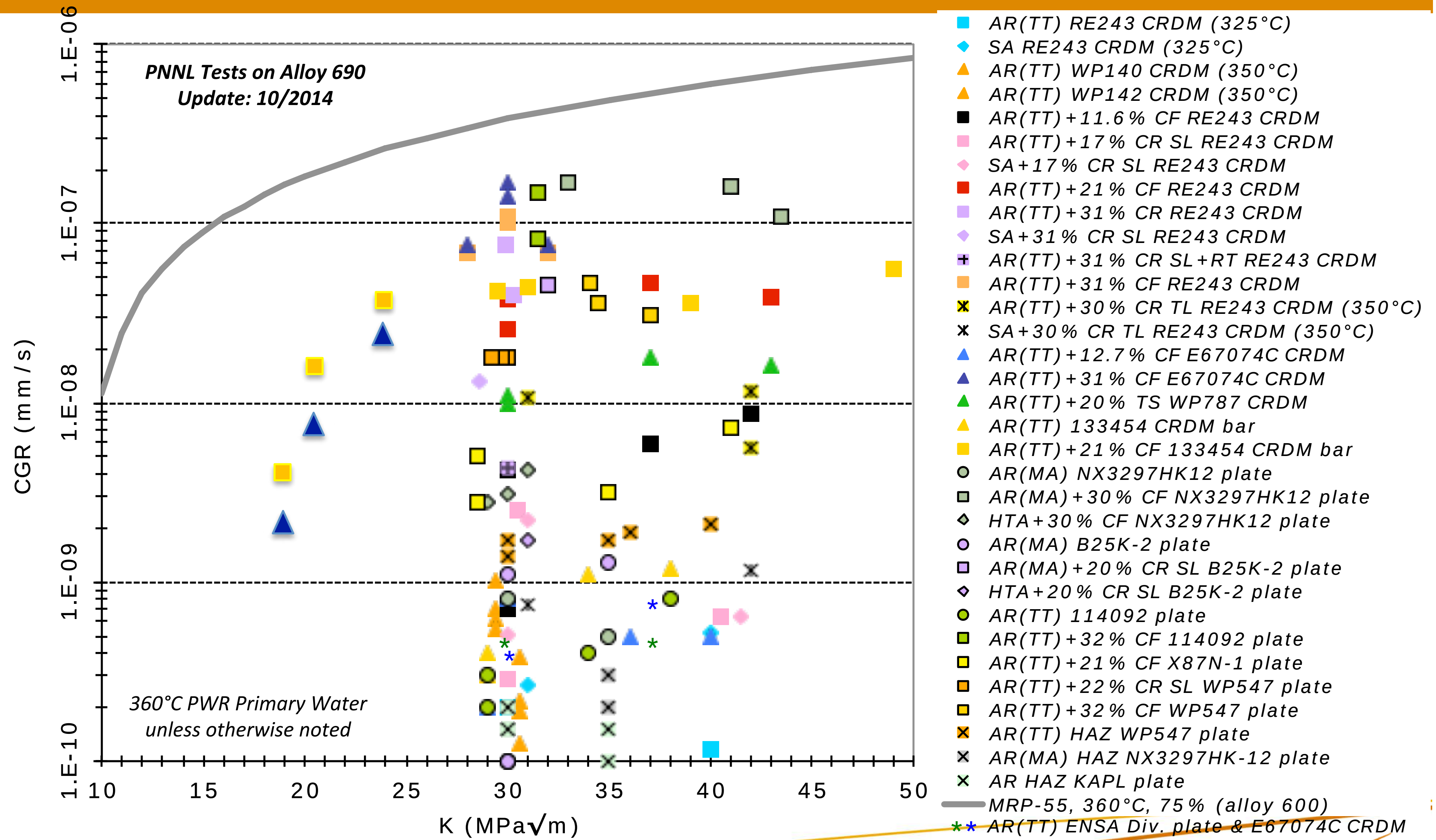
SCC Crack Growth Response for the 31%CW Alloy 690TT CRDM Material



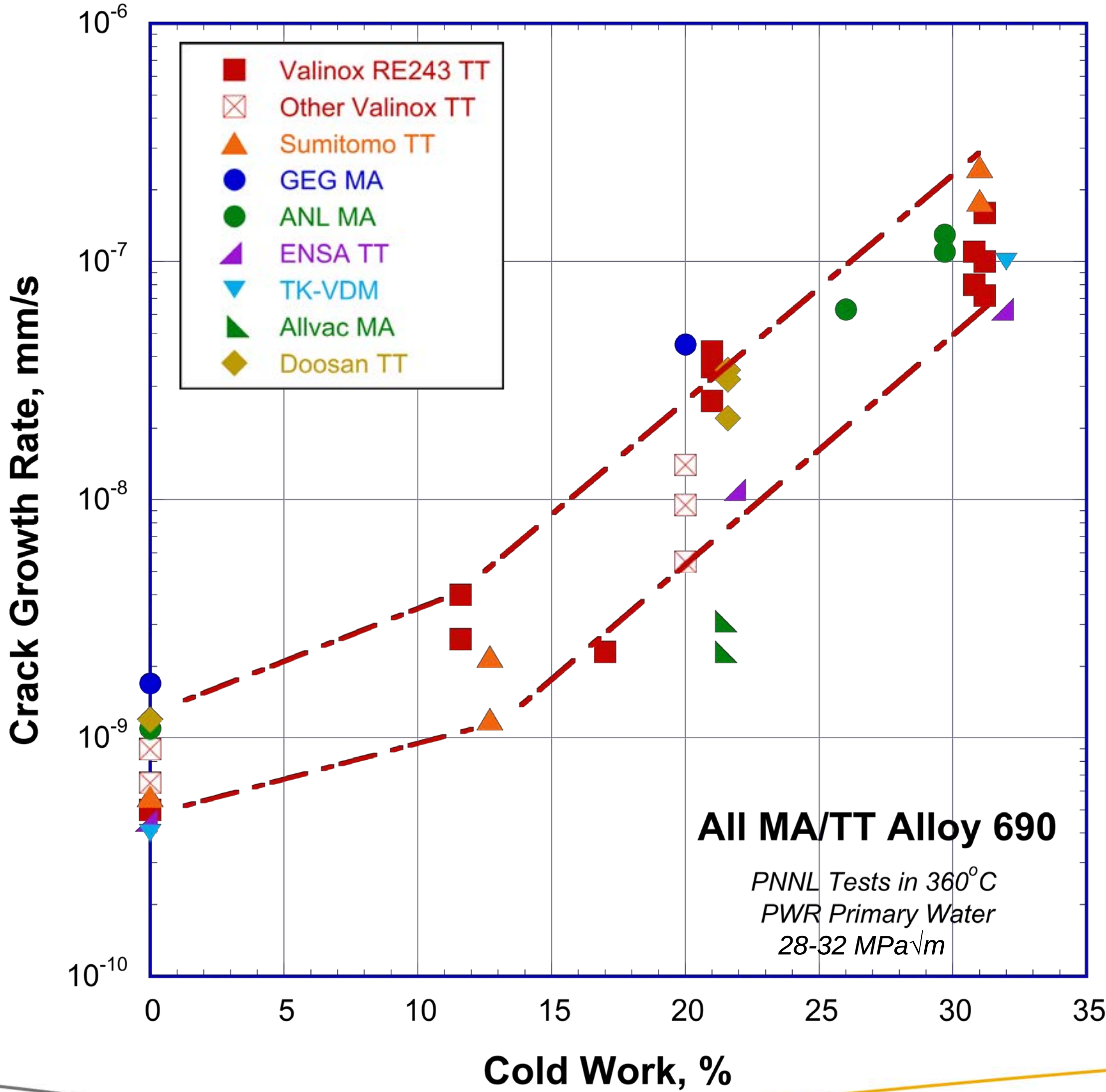
Stress intensity effects on SCC growth rates were evaluated on two alloy 690 CRDM heats cold forged to a 31% reduction. Rates discovered to increase from ~4x10-9 mm/s at ~19 MPa√m to ~1x10-7 mm/s at 30 MPa√m.

Summary of PNNL Alloy 690 Measurements of SCC Growth Rates



Full spectrum of measured SCC growth rates illustrating significant effect of cold work from 0-32% reduction.

Summary of PNNL Alloy 690 Measurements of SCC Growth Rates



Consistent increase in measured SCC growth rates as a function of cold work for alloy 690 materials in the as-received MA or TT condition. Data on these heats suggest a transition in SCC susceptibility for materials cold worked to >15% reduction. However, additional testing is recommended at low cold-work levels.

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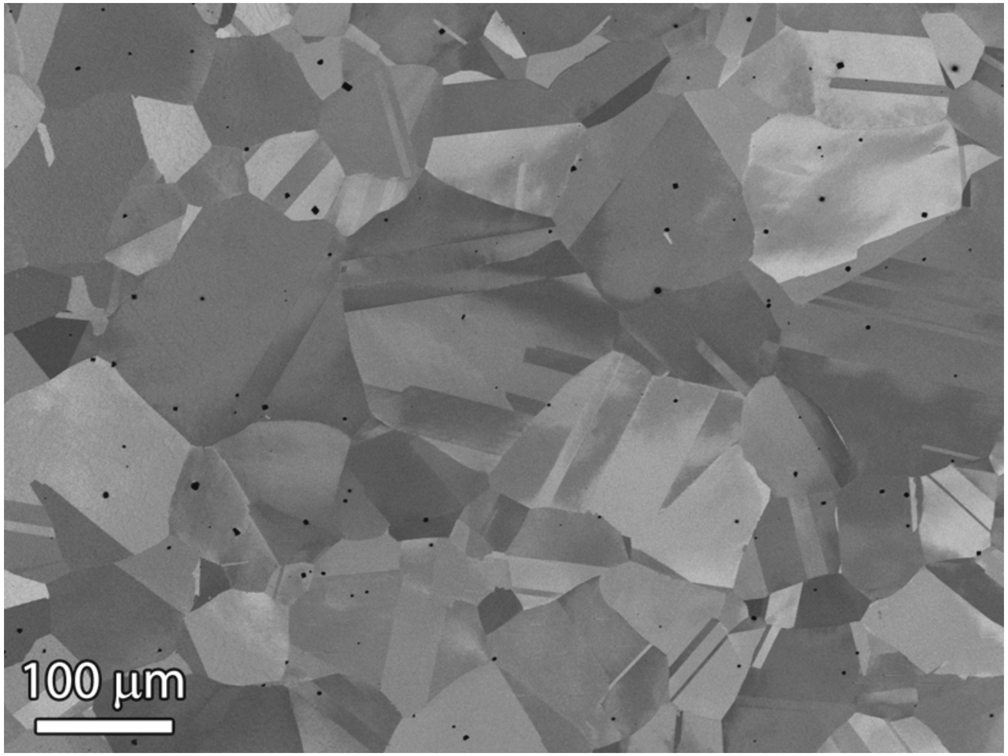
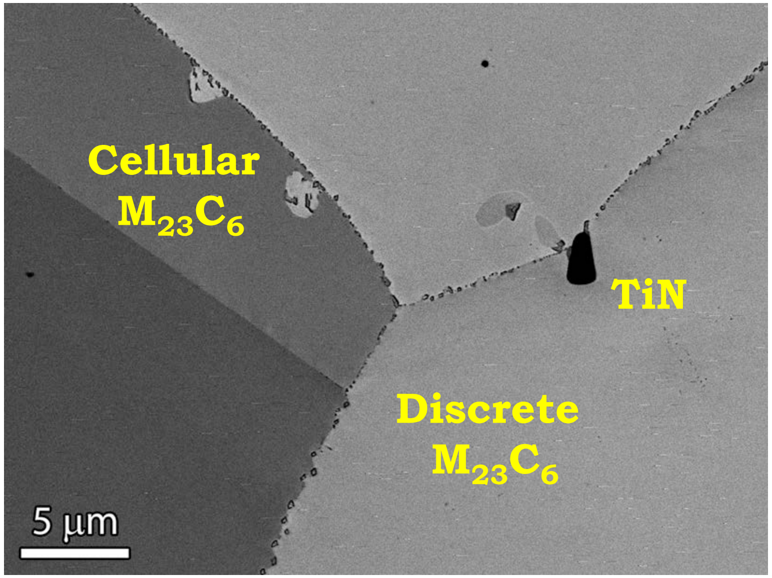
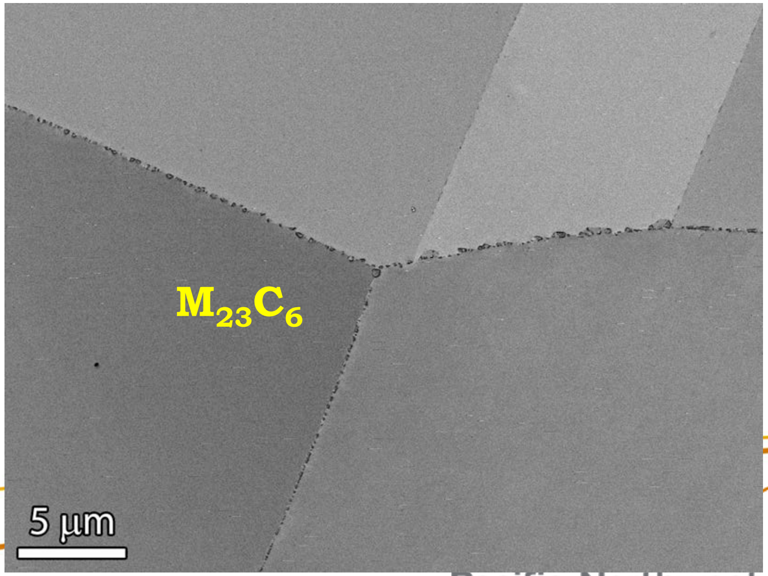
PNNL Alloy 690 Characterization Activities

- ***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*

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



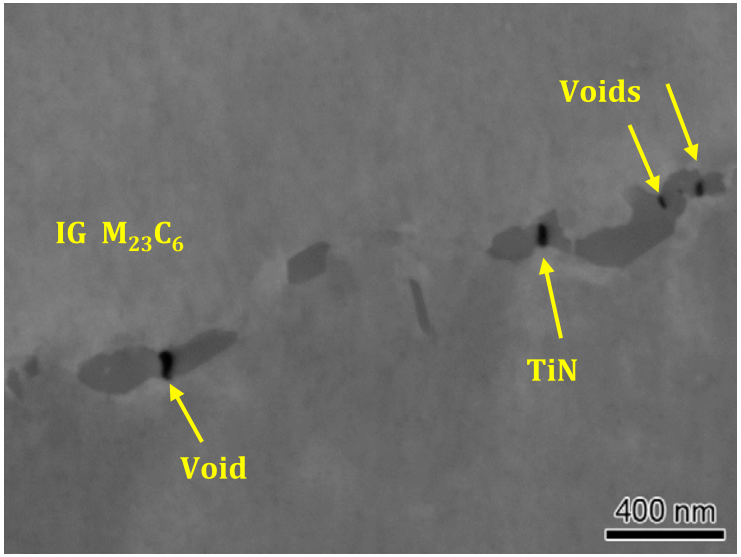


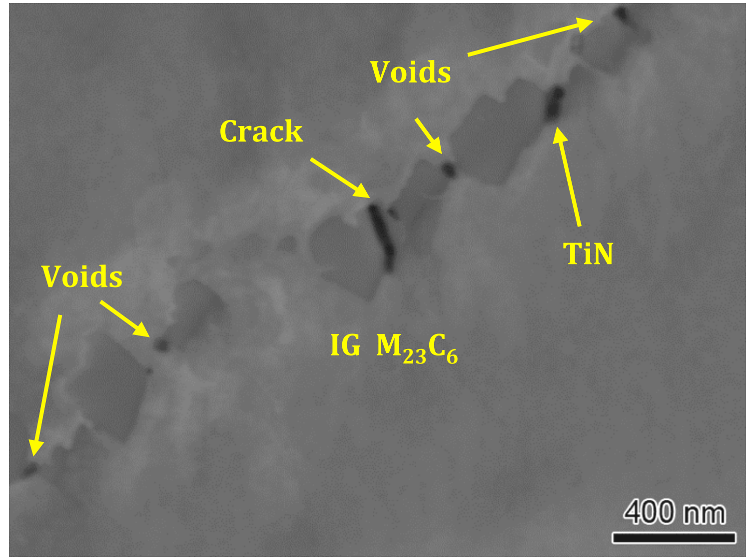


SEM backscatter images show general microstructure and grain boundary carbides in alloy 690TT CRDM tubing heat RE243. High density of nearly continuous, grain boundary carbides with well-spaced Ti nitrides.

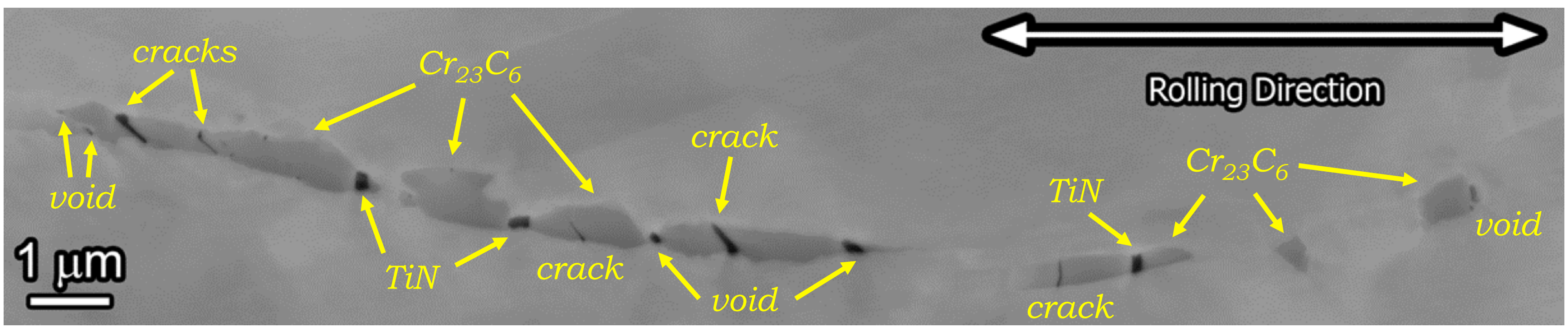
Examples of Grain Boundary Voids and Cracked Carbides in Highly CR Alloy 690

31%CR CRDM Tube



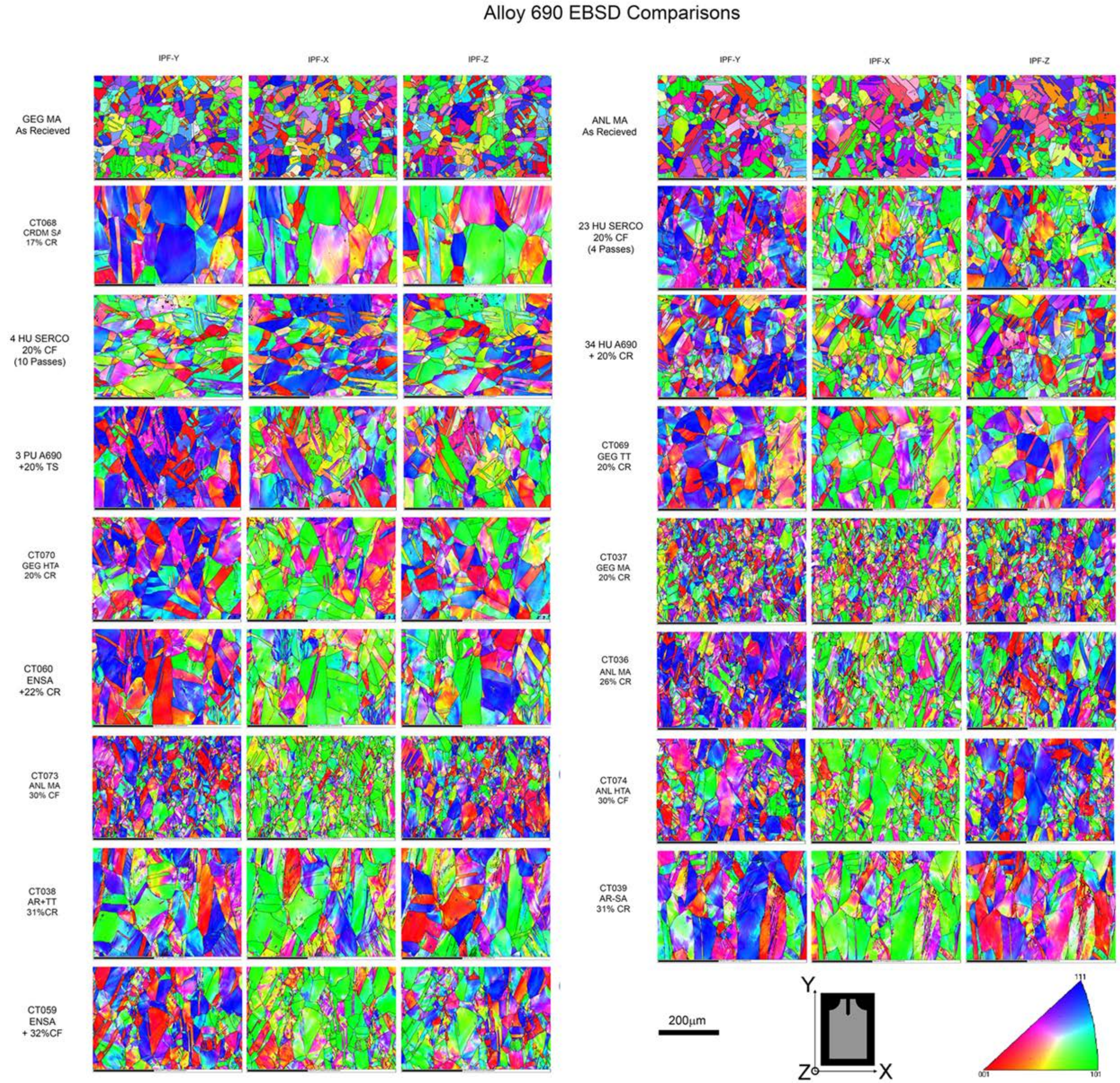


26%CR Plate



SEM examinations at higher magnifications and different imaging conditions along with EDS composition maps can identify most features.
No correlation found between CW voids/cracks and SCC response.

EBSD Measurements on Cold Worked Alloy 690 Materials



EBSD measurements including inverse pole figures (shown) and misorientation densities have been performed on crack growth compact tension specimens. Regions analyzed are from the crack growth plane where SCC propagation rates were obtained.

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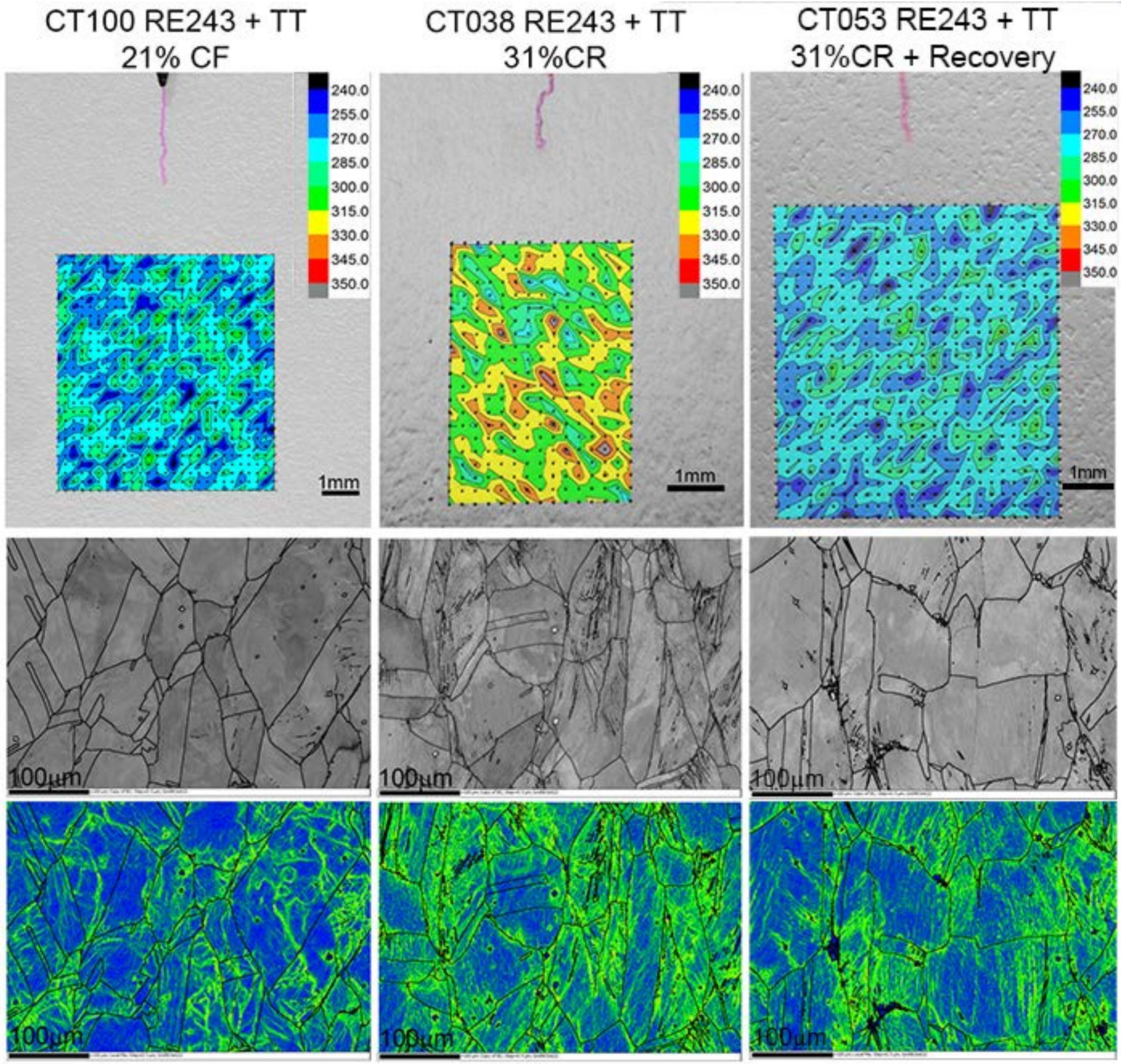
Hardness and EBSD Measurements on Cold-Worked Alloy 690 Materials



Hardness maps have been obtained on crack growth compact tension specimens after EBSD measurements. Regions analyzed are from the crack growth plane where SCC propagation rates were obtained.

Examples show that the recovery heat treatment decreases strain density and hardness if 31%Cw material approaching the 21%CW material.

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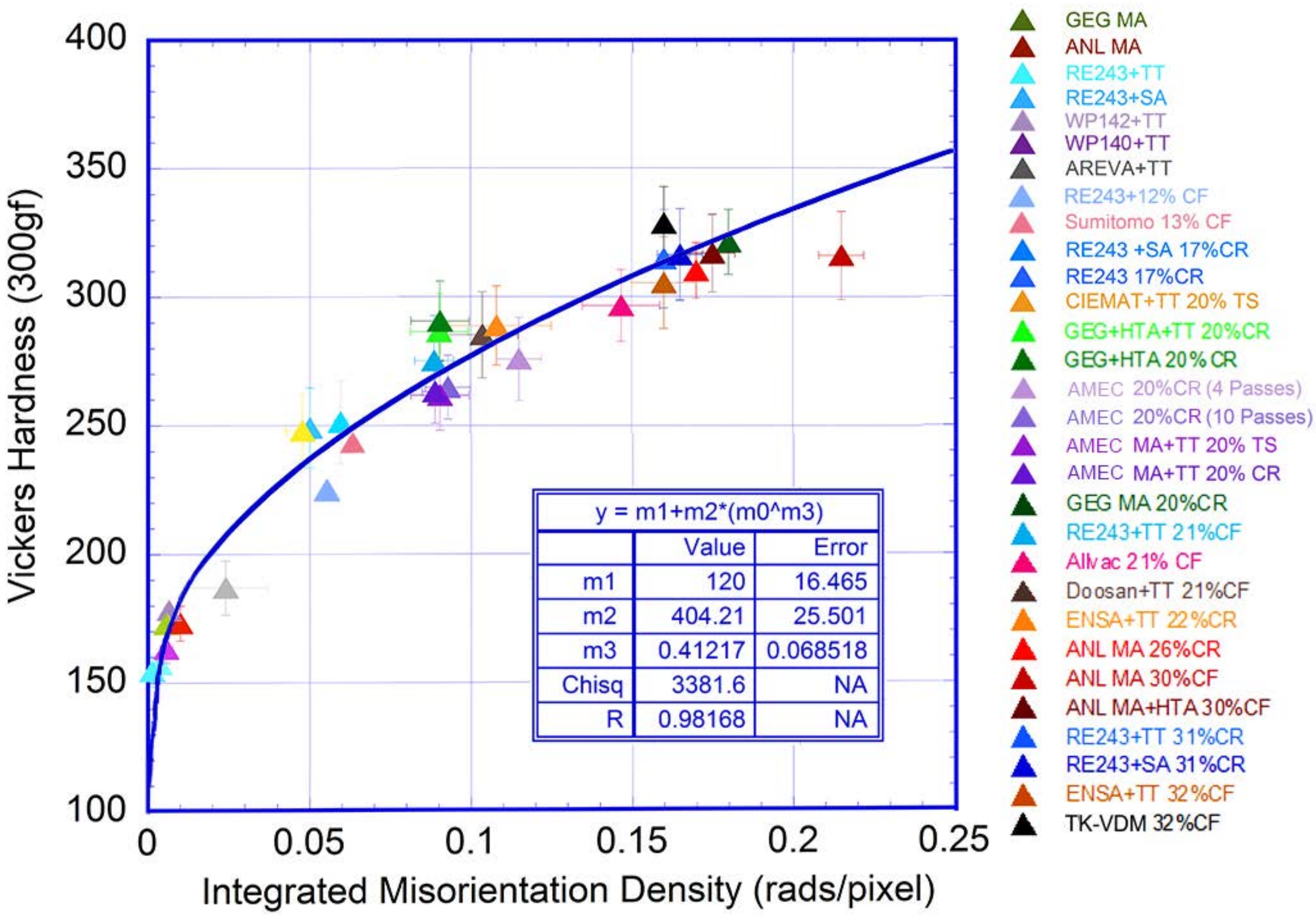
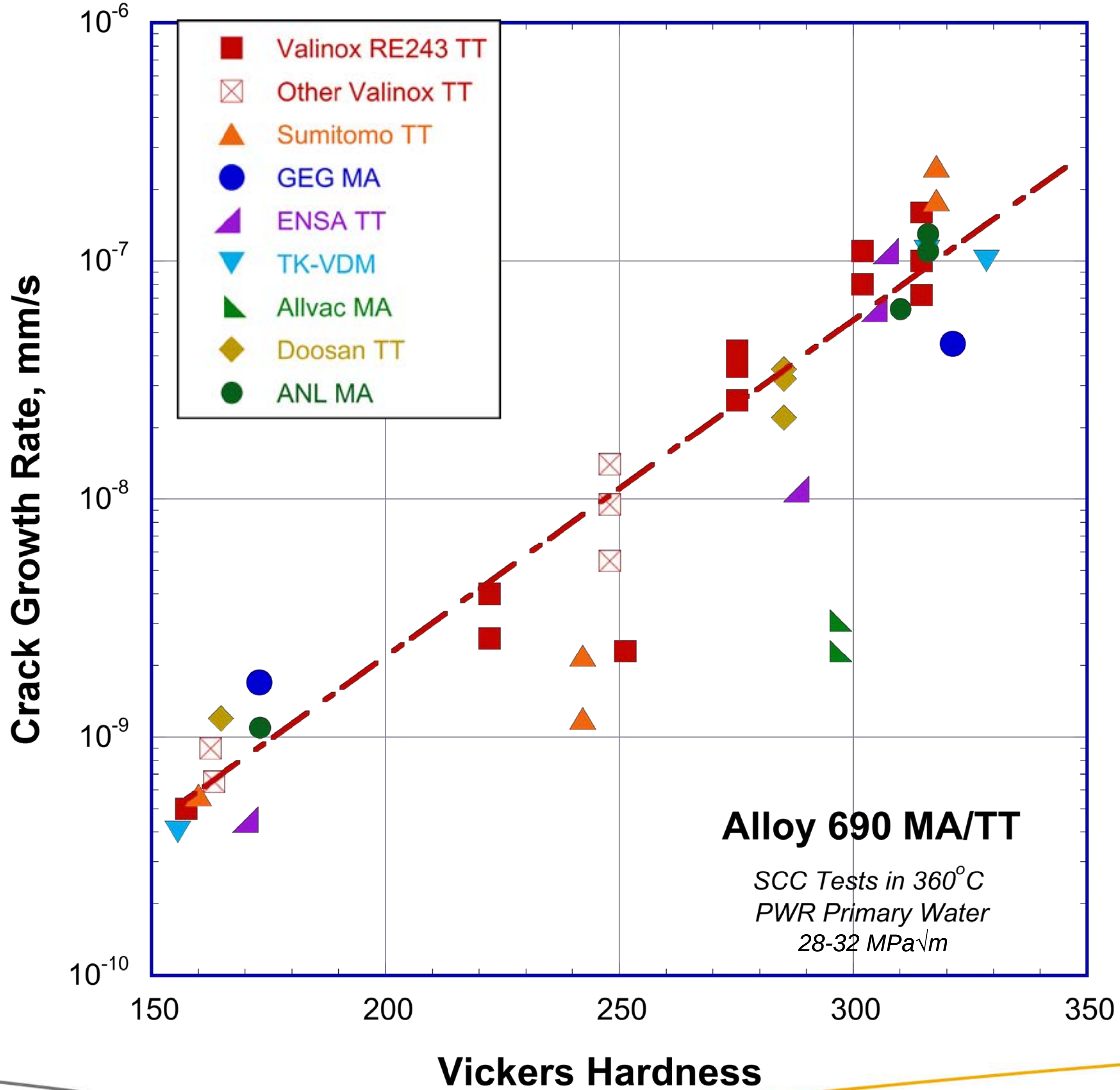
Hardness and EBSD Misorientation Measurements in CW Alloy 690 Materials



Measured hardness plotted versus EBSD misorientation density showing expected square root dependence and indicating a strain hardening exponent (m3) consistent with that reported for alloy 690.

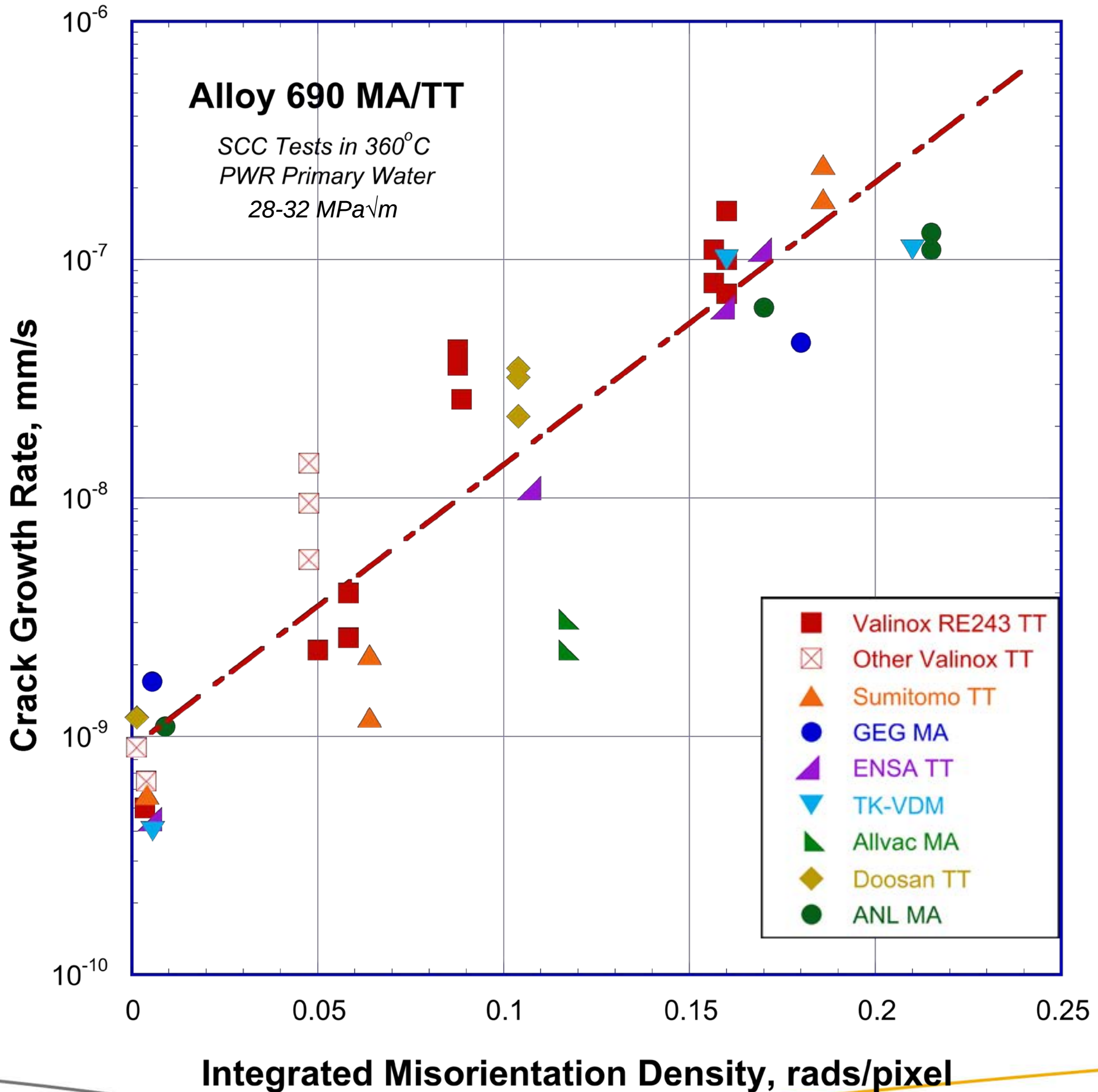
Correlations Between Hardness and SCC Growth Rates



Results on CRDM, plate, bar and billet heats indicate low SCC growth rates (<~10^{-8} mm/s) at hardness values below ~240 kg/mm^2.

Measured SCC rates suggest a continuous increase in susceptibility with hardness for cold-worked alloy 690 materials in the as-received MA or TT condition. However, some heat-to-heat differences are present.

Correlations Between EBSD Misorientation Density and SCC Growth Rates



Measured SCC rates show a continuous increase in susceptibility with EBSD-measured matrix strain for cold-worked alloy 690 materials in the as-received MA or TT condition. However, some heat-to-heat differences are present.

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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 ($\geq$~15%CW) condition. Significant heat-to-heat and material condition differences in SCC susceptibility are observed for 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.*
- *Average EBSD-measured strains and hardness values correlate well to measured SCC growth rates in the alloy 690 materials. Current data indicates that matrix strength and deformation structures near grain boundaries control IGSCC susceptibility.*

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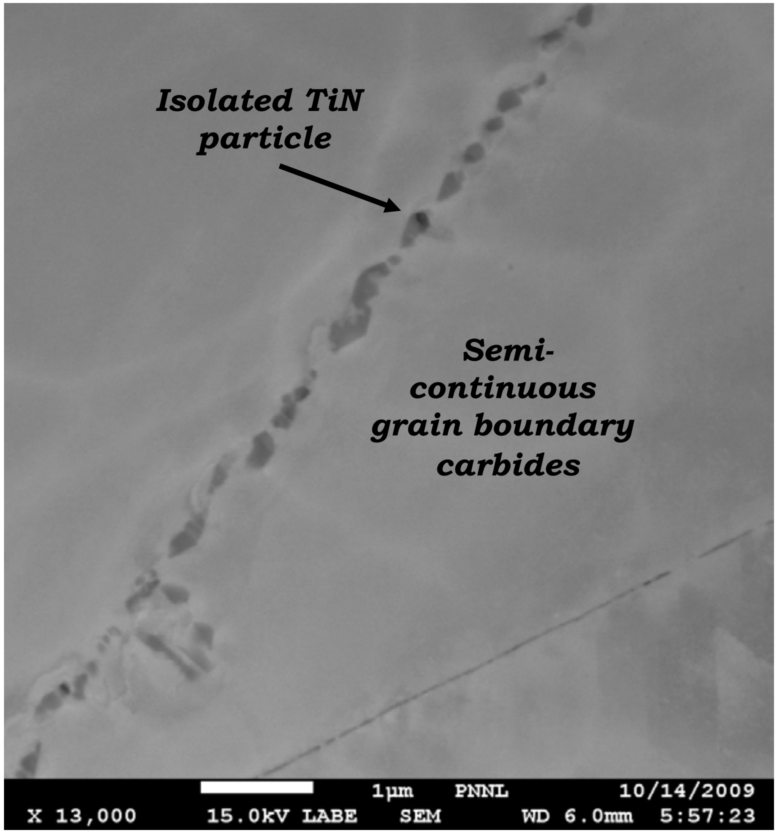
PNNL Alloy 690 Test Summary
Effect of High-Temperature Anneal (HTA)

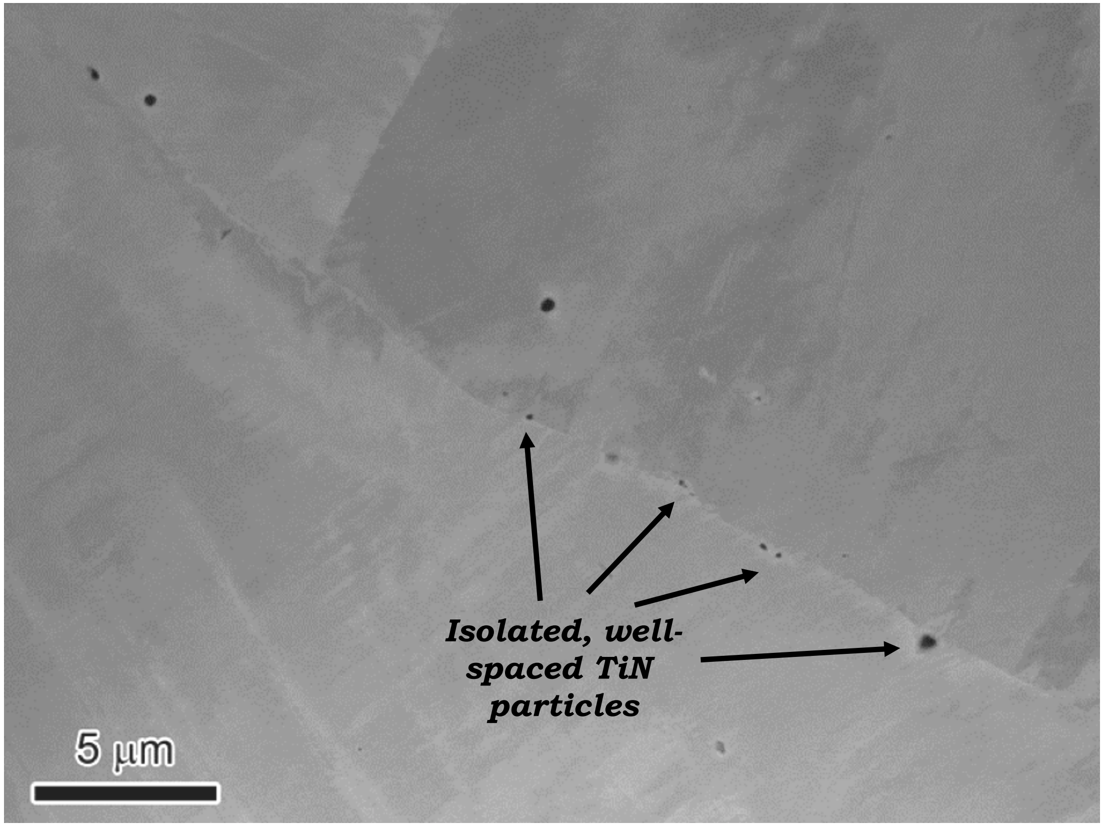
- ***Alloy 690 CRDM***
  - *As-received TT heats: Valinox RE243, WP140 and WP142; Doosan 133454; Sumitomo E67074C*
  - *As-received TT (high density of IG Cr carbides, Cr depletion) versus HTA/SA for Valinox RE243 + 0%, 17%CR, 30%CR, 31%CR; post-CW recovery anneal: 31%CR alloy 690TT + 700°C/1h*
  - *As-received TT + 12%CF, 21%CF & 30%CF Valinox RE243; 20%TS Valinox (WP787); 13%CF & 30%CF Sumitomo; 21%CF Doosan*
- ***Alloy 690 Plate/Bar Materials***
  - *As-received MA heats: ANL (NX3297HK12), GEG (B25K), TK-VDM (114092), ENSA Divider Plate (TT)*
  - *MA versus HTA for 30%CF ANL and 20%CR GEG heats*
  - *As-received + 21%CF Allvac (X87N-1), 22%CR & 30%CF ENSA (SP547), 26%CR ANL, 32%CF TK-VDM*
  - *CF versus HF specimens from GEG: 32%CF and 30%HF ENSA; 33%CF and 30%HF TK-VDM*

Effect of High-Temperature Anneal (HTA) on Grain Boundary Microstructures in Alloy 690

Alloy 690TT CRDM

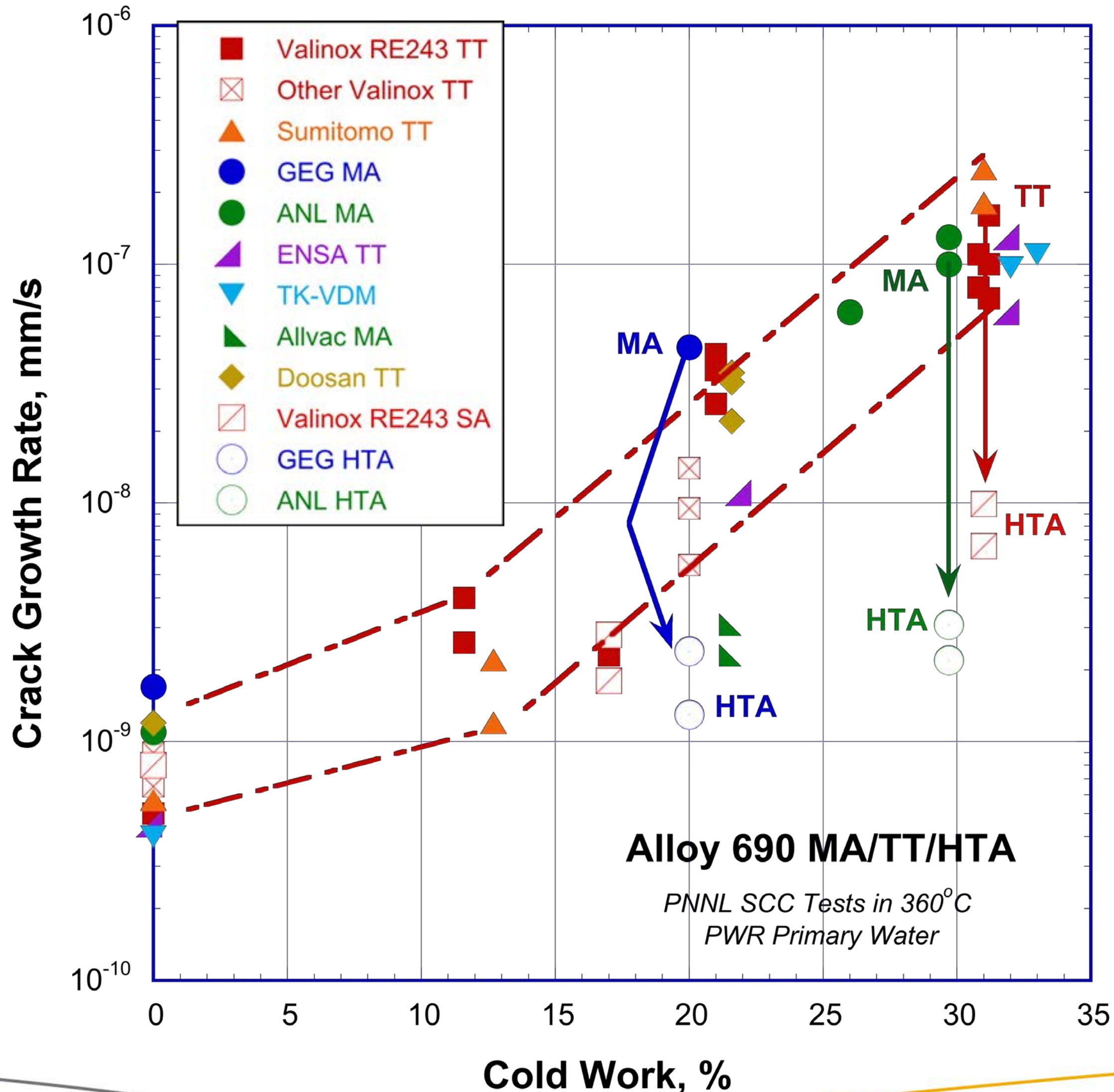


Alloy 690TT + HTA



High-temperature anneal at 1100°C and water quench removed nearly all grain boundary carbides, only isolated TiN particles remain. Overall average grain size is also slightly increased.

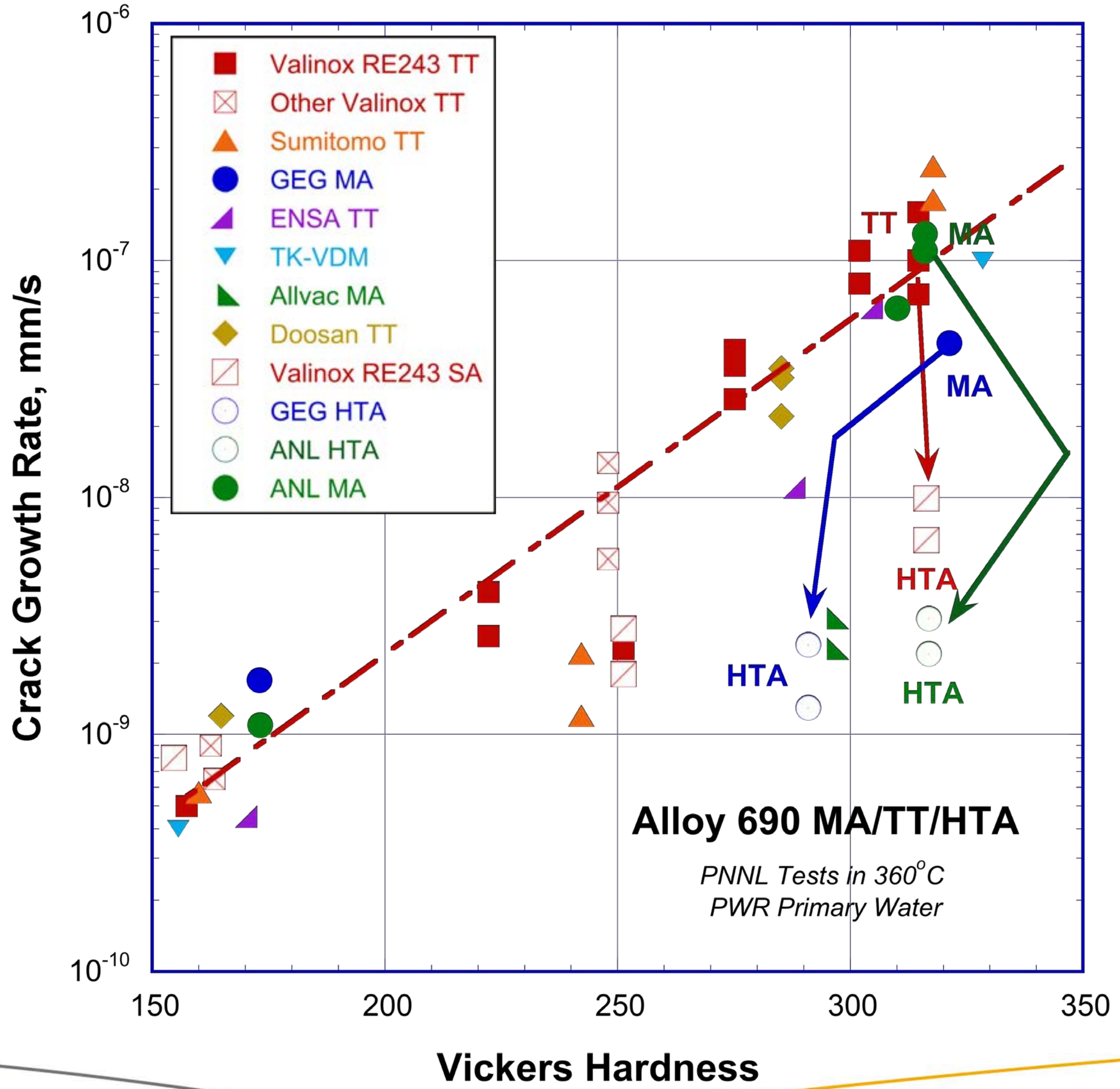
Summary of PNNL Alloy 690 SCC Growth Rates Measurements



The influence of an initial high temperature anneal (HTA) at 1100C and rapid water quench on SCC response after cold work was evaluated for three alloy 690 heats.

Measured SCC growth rates were lower in the highly cold-worked HTA materials by 10-50X.

Correlations Between Hardness and SCC Growth Rates



The HTA pretreatment did not significantly alter hardness after extensive cold work. As a result, the reduced SCC susceptibility for the cold-worked HTA materials cannot be explained by differences in matrix hardness.

Primary effects of HTA was to remove grain boundary carbides and slightly increase grain size.

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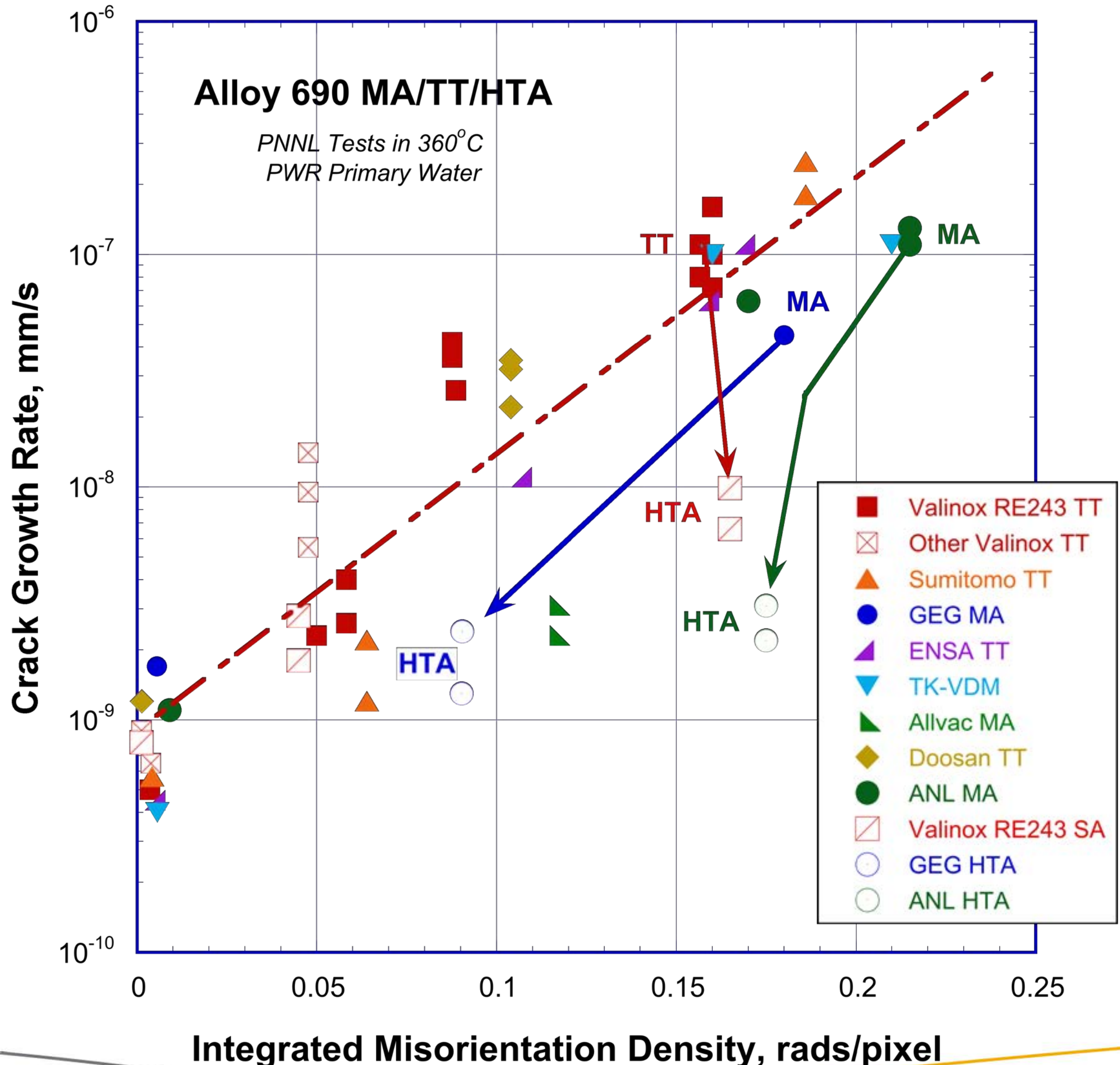
Correlations Between EBSD-Measured Matrix Strain and SCC Growth Rates



EBSD-measured strain densities for cold-worked HTA materials were lower for two heats and similar for one. As a result, the reduced SCC susceptibility for the HTA materials cannot be explained by differences in the matrix strain densities.

Primary effect of HTA on microstructure was to remove grain boundary carbides and slightly increase grain size.

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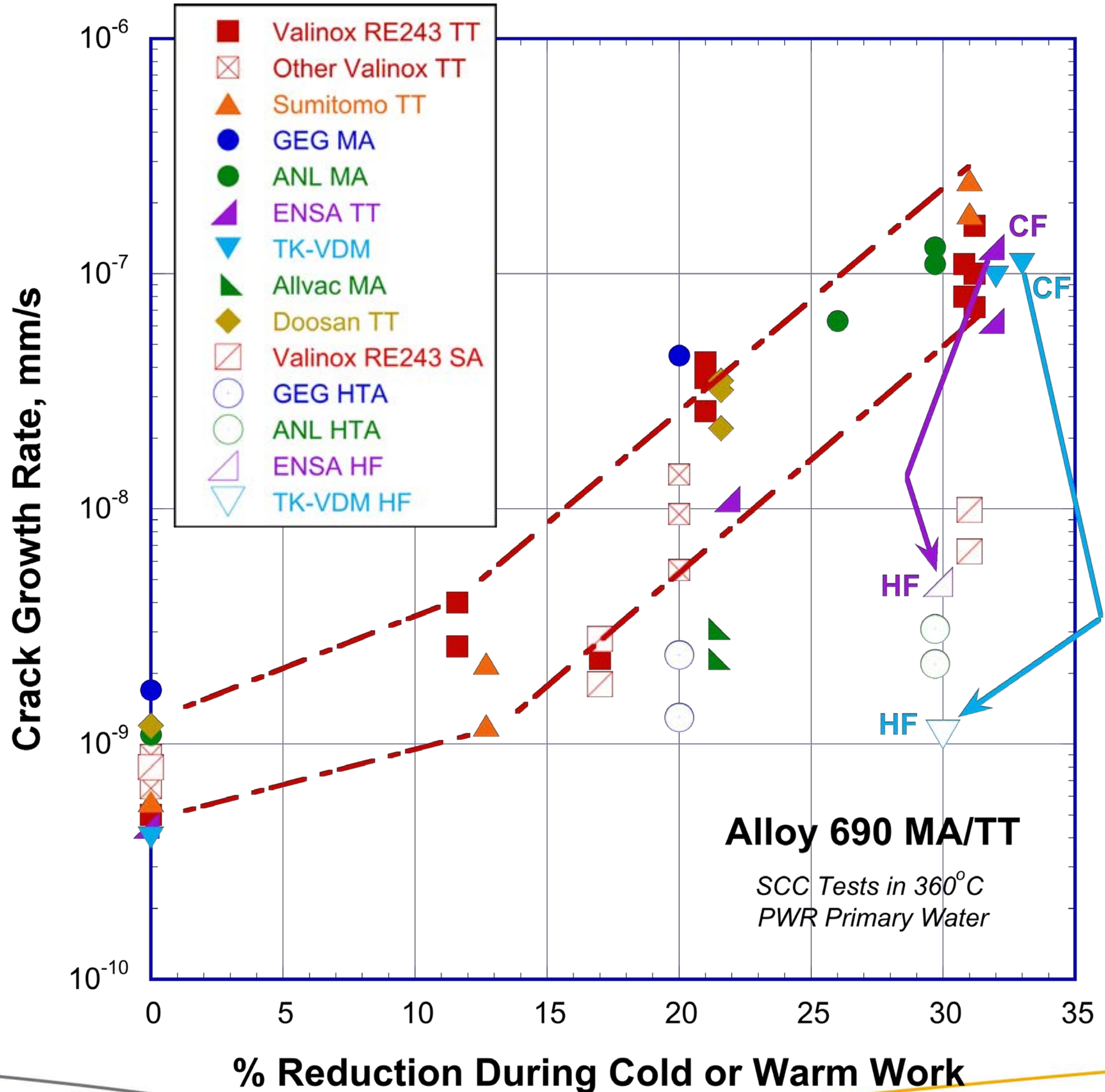
Effect of High-Temperature Anneal on IGSCC of Cold-Worked Alloy 690

- *Initial high temperature anneal and quench improves SCC resistance in highly CW alloy 690 heats. Primary microstructural changes are the removal of most grain boundary carbides and slightly increasing the overall grain size.*
- *Hardness and EBSD-measured matrix strain measurements reveal similar values for the AR+30%CW and AR+HTA+30%CW materials. Therefore, the HTA pre-treatment did not significantly effect the development of matrix strain and strength during subsequent cold work.*
- *Most likely reason for improved SCC resistance in cold-worked HTA materials is that the change in IG carbide microstructure modified the development of local strain near grain boundaries.*

PNNL Alloy 690 Test Summary
Effect of Hot versus Cold Forging

- ***Alloy 690 CRDM***
  - *As-received TT heats: Valinox RE243, WP140 and WP142; Doosan 133454; Sumitomo E67074C*
  - *As-received TT (high density of IG Cr carbides, Cr depletion) versus HTA/SA for Valinox RE243 + 0%, 17%CR, 30%CR, 31%CR; post-CW recovery anneal: 31%CR alloy 690TT + 700°C/1h*
  - *As-received TT + 12%CF, 21%CF & 30%CF Valinox RE243; 20%TS Valinox (WP787); 13%CF & 30%CF Sumitomo; 21%CF Doosan*
- ***Alloy 690 Plate/Bar Materials***
  - *As-received MA heats: ANL (NX3297HK12), GEG (B25K), TK-VDM (114092), ENSA Divider Plate (TT)*
  - *MA versus HTA for 30%CF ANL and 20%CR GEG heats*
  - *As-received + 21%CF Allvac (X87N-1), 22%CR & 30%CF ENSA (SP547), 26%CR ANL, 32%CF TK-VDM*
  - *CF versus HF specimens tested by Peter Andresen at GEG: 32%CF and 30%HF ENSA; 33%CF and 30%HF TK-VDM*

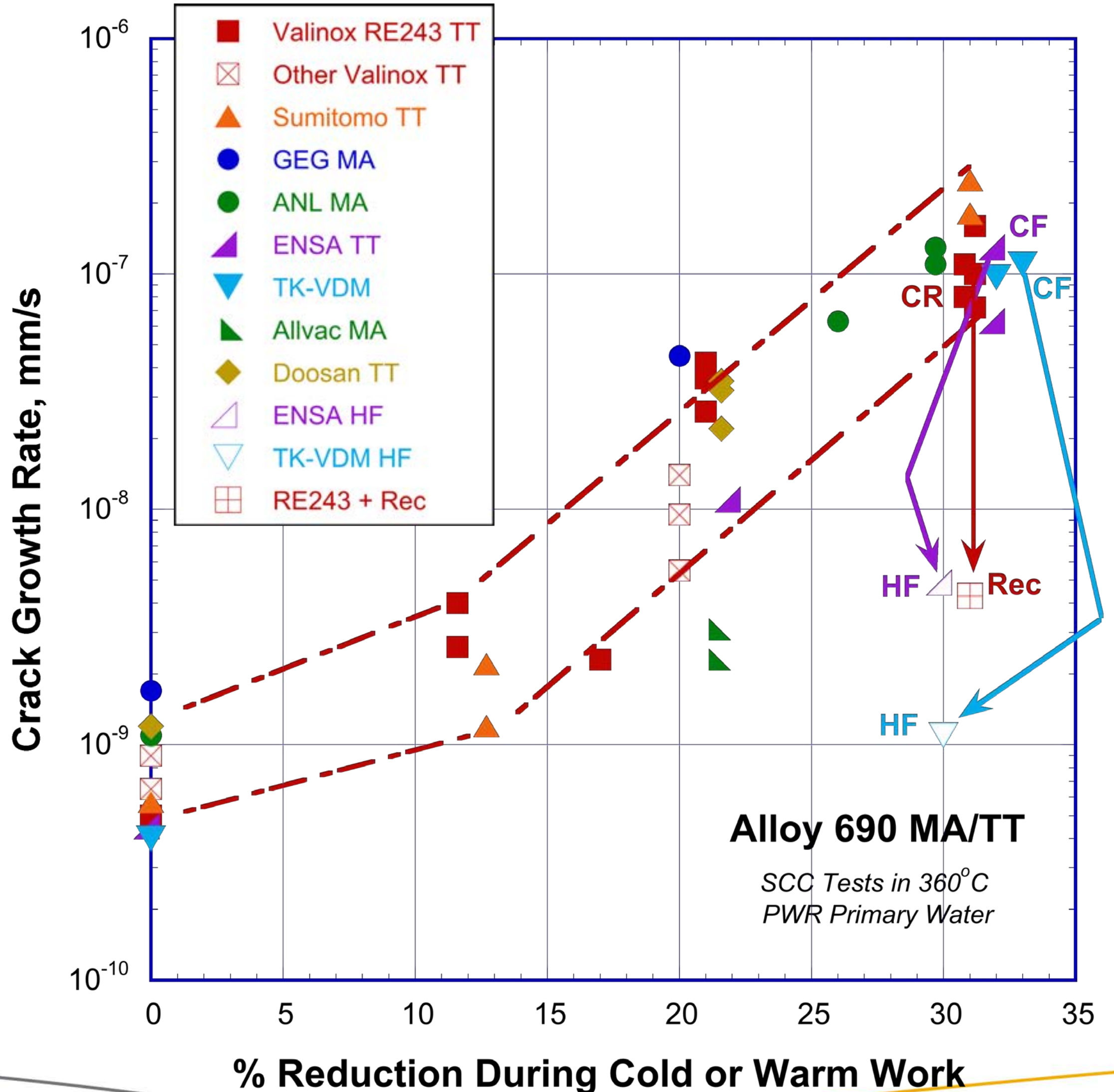
Summary of Alloy 690 Measurements of SCC Growth Rates



The influence of hot forging at 700C versus cold forging has been evaluated by Peter Andresen at GEG. Measured SCC growth rates were lower in the HF materials by 25-100X.

Hardness and EBSD characterizations have been performed at PNNL to help understand reasons for differences in SCC susceptibility.

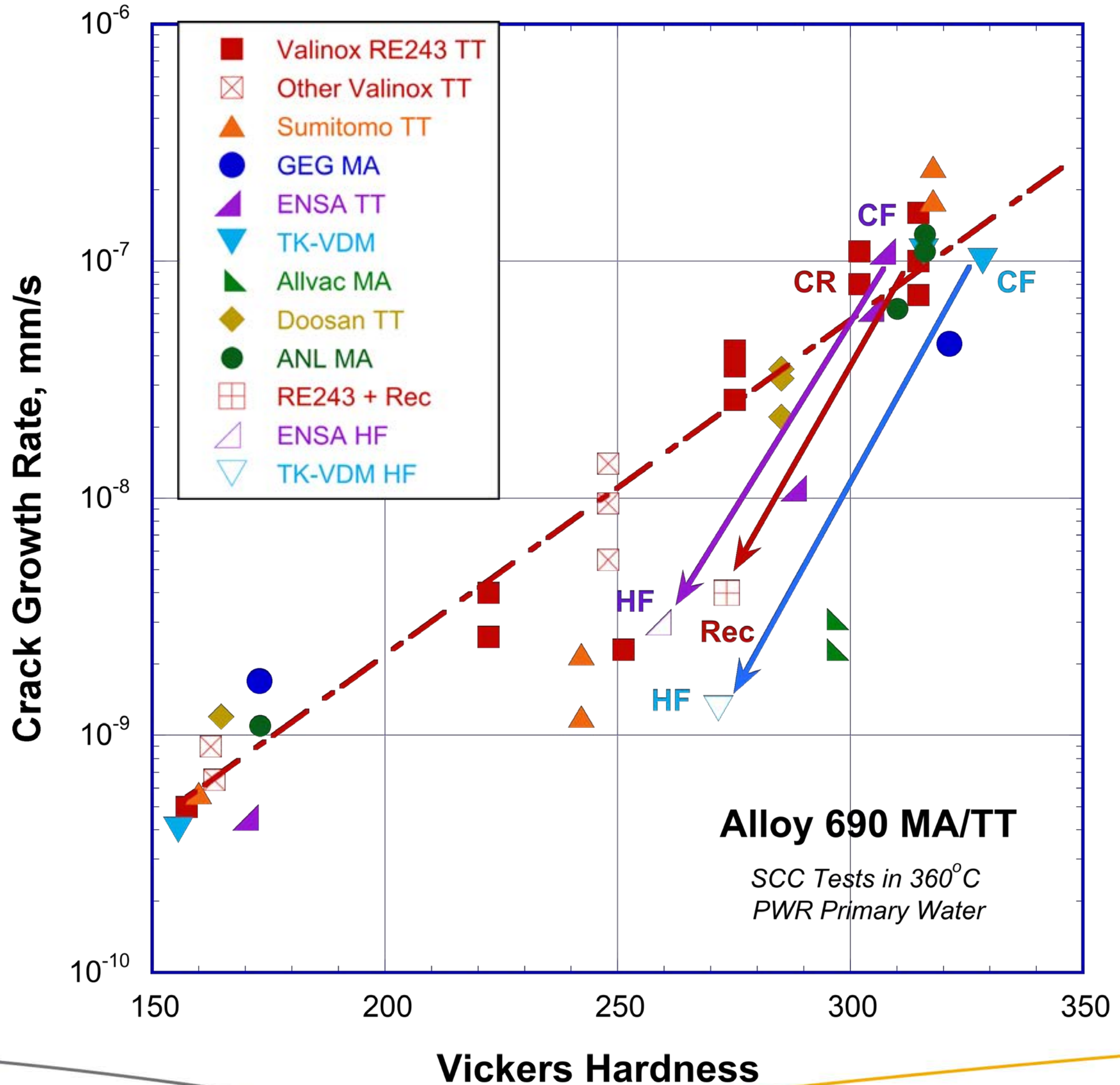
Summary of Alloy 690 Measurements of SCC Growth Rates



An alloy 690TT CRDM heat was cold rolled to 31% reduction and then given a 700C recovery anneal for 1 hour. This resulted in measured SCC growth rates than were ~25X lower than the cold-rolled material.

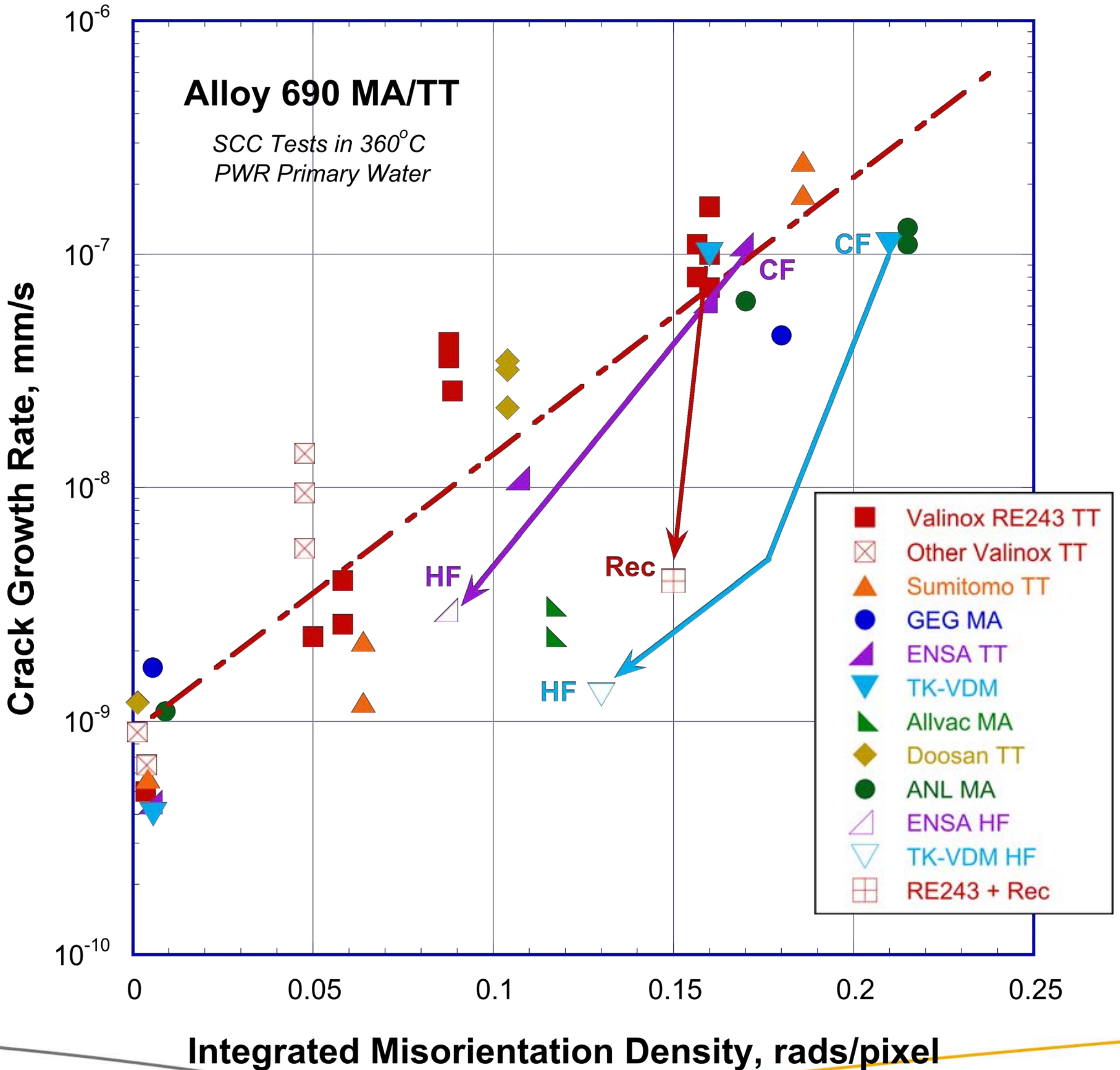
The decrease in SCC susceptibility was similar to the observed effect of hot forging at 700C.

Correlations Between Hardness and SCC Growth Rates



Hot forging at 700C results in significantly lower hardness than cold forging to a ~30% reduction. This decrease in hardness was consistent to giving the cold-worked material a short recovery anneal (RA) at 700C. However, HF and RA materials exhibit SCC growth rates somewhat below the general correlation with hardness for as-received alloy 690.

Correlations Between EBSD-Measured Matrix Strain and SCC Growth Rates

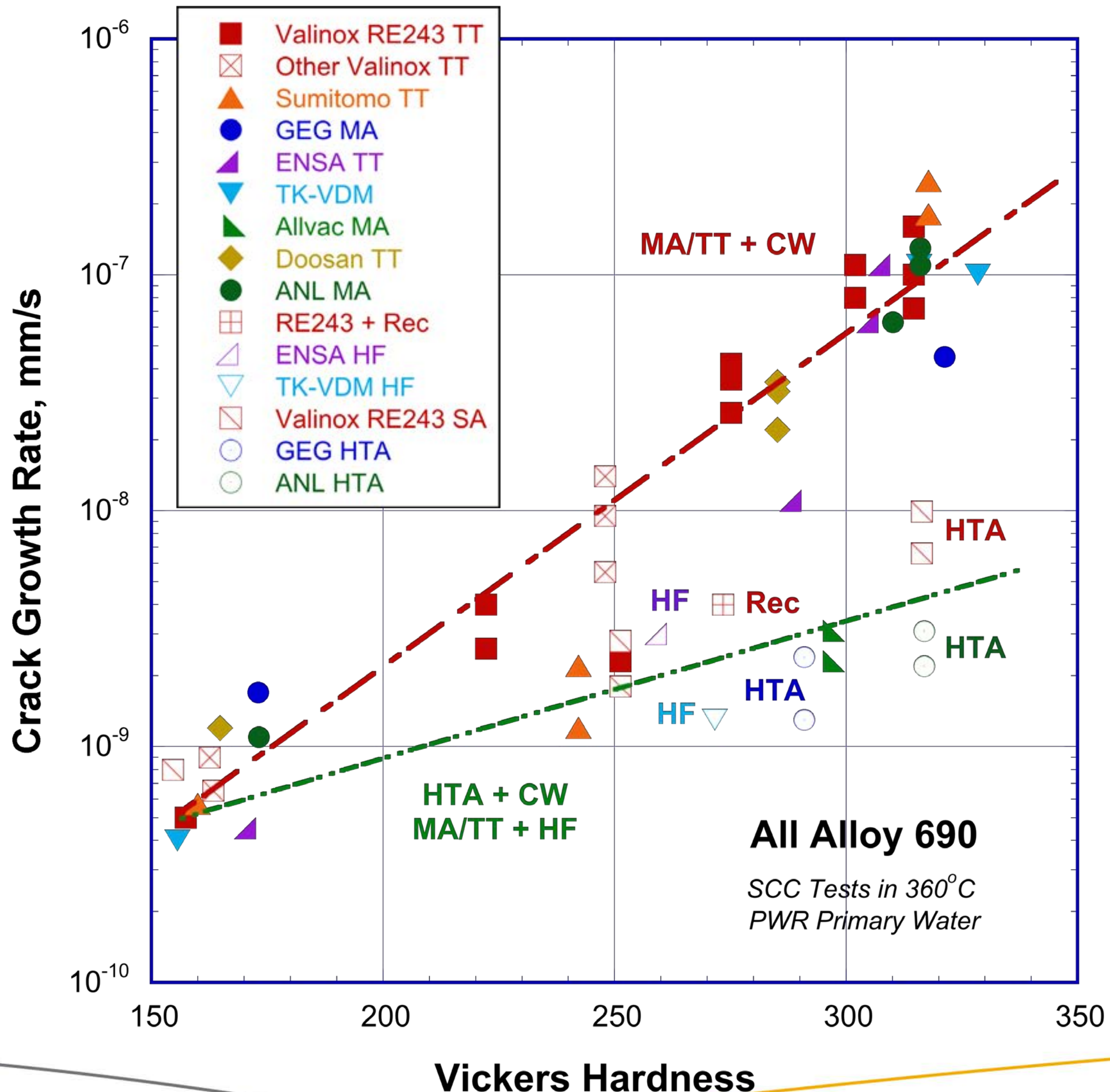


Hot forging at 700C results in significantly lower matrix strain than cold forging to a ~30% reduction. The short recovery anneal (RA) at 700C also reduced matrix strain, but to a smaller degree. However, HF and RA materials exhibit SCC growth rates below that expected from the general correlation with EBSD misorientation density for as-received alloy 690.

Effect of Cold versus Hot Forging on Hardness, Strain and IGSCC of Alloy 690

- *SCC experiments at GE Global Research showed that hot forging at 700C to a reduction of ~30% produced a much lower SCC growth rate than cold forging.*
- *Hardness and EBSD-measured matrix strain measurements at PNNL reveal that hot forging does produce lower values than cold forging. This is consistent with the SCC response, however it seems likely that lower grain boundary strains are also created during hot forging and impact SCC behavior.*
- *A recovery anneal at 700C was found to have a similar effect on hardness, matrix strain and SCC growth rates when given to a highly cold-worked material as seen for the hot forged materials.*

Correlations Between Hardness and SCC Growth Rates



Correlation between measured SCC growth rates and hardness are different for the highly cold-worked HTA and hot-forged TT materials than for the cold-worked MA/TT materials. Therefore, something beyond matrix strength is influencing IGSCC susceptibility. Differences in localized grain boundary strain appears the most likely cause.

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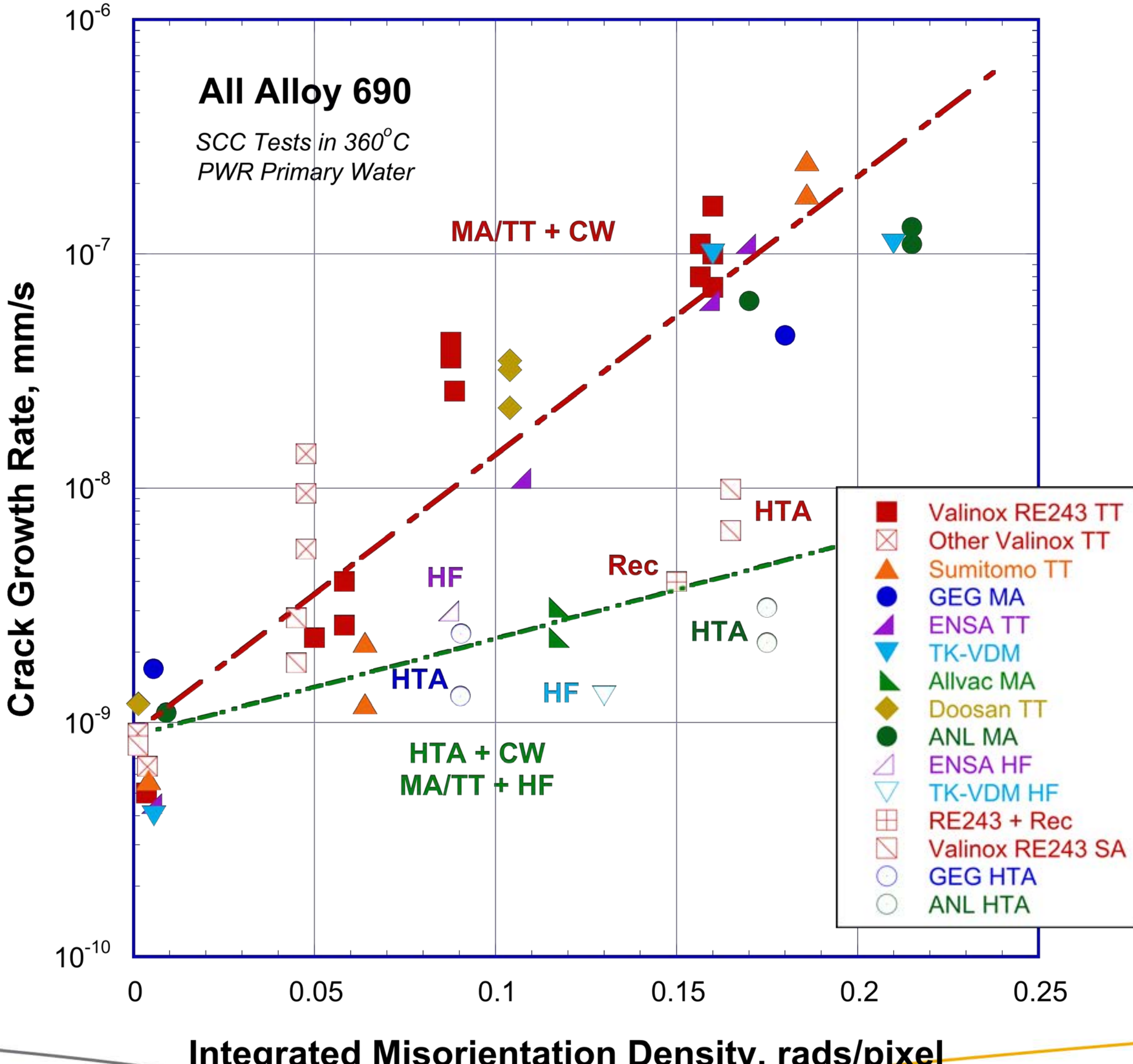
Correlations Between Misorientation Density and SCC Growth Rates



Correlation between measured SCC growth rates and EBSD-measured matrix strains are different for the highly cold-worked HTA and hot-forged TT materials than for the cold-worked MA/TT materials. Therefore, something beyond matrix strength is influencing IGSCC susceptibility. Differences in localized grain boundary strain appears the most likely cause.

Cold Work Effects on Stress Corrosion Crack Growth of Alloy 690 Materials

- *This presentation highlights research results at PNNL on cold-worked alloy 690 materials over the last ~7 years including SCC growth rate measurements for the U.S. NRC and more recent material characterizations co-funded by Rolls Royce.*
- *Most of these results are summarized in much more detail in an NRC report that is currently in press: NUREG/CR-7103 Volume 3.*