Grain Boundary Damage in Alloy 690 after One Dimensional Cold Rolling

Preliminary Observations

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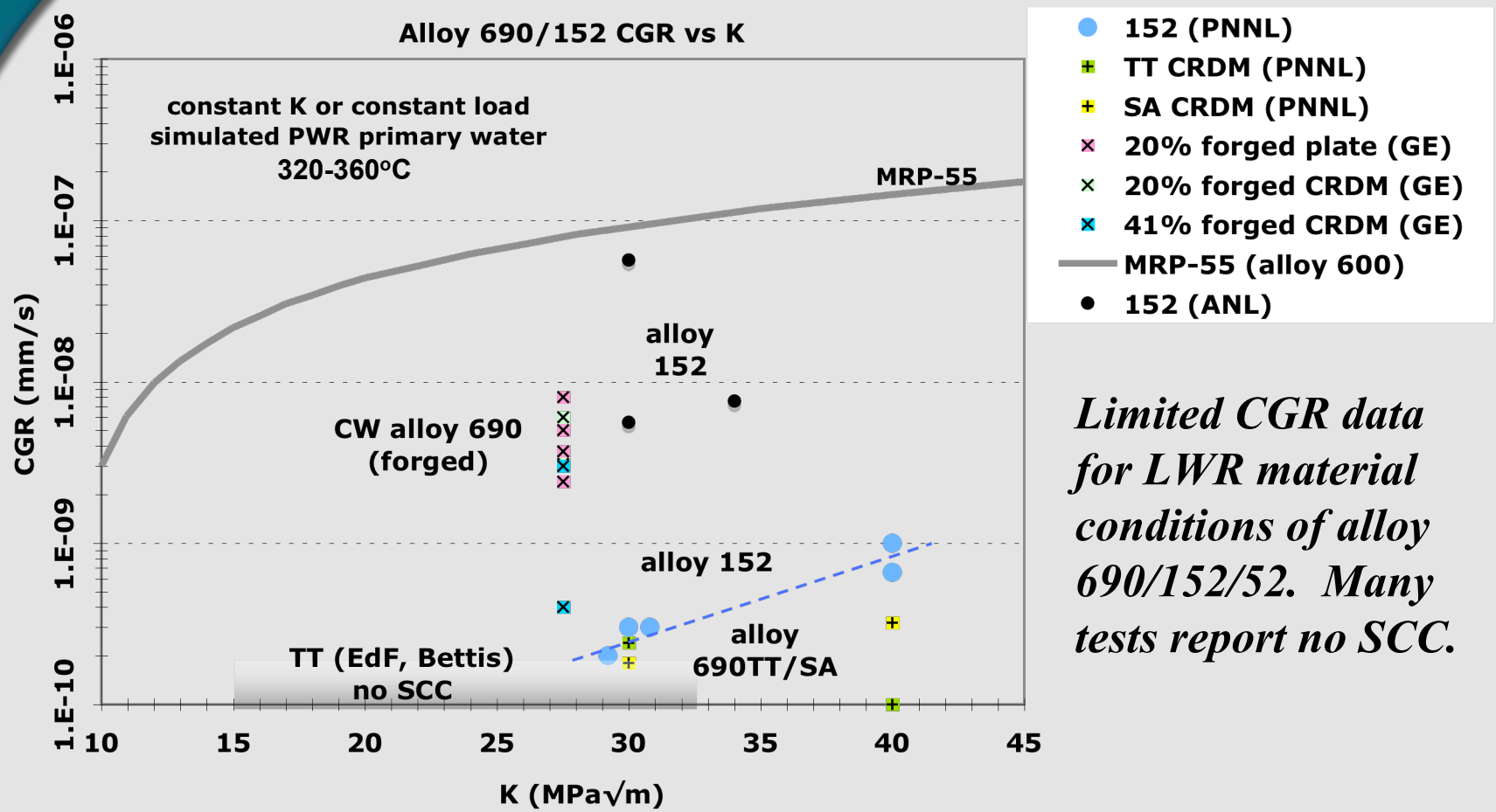
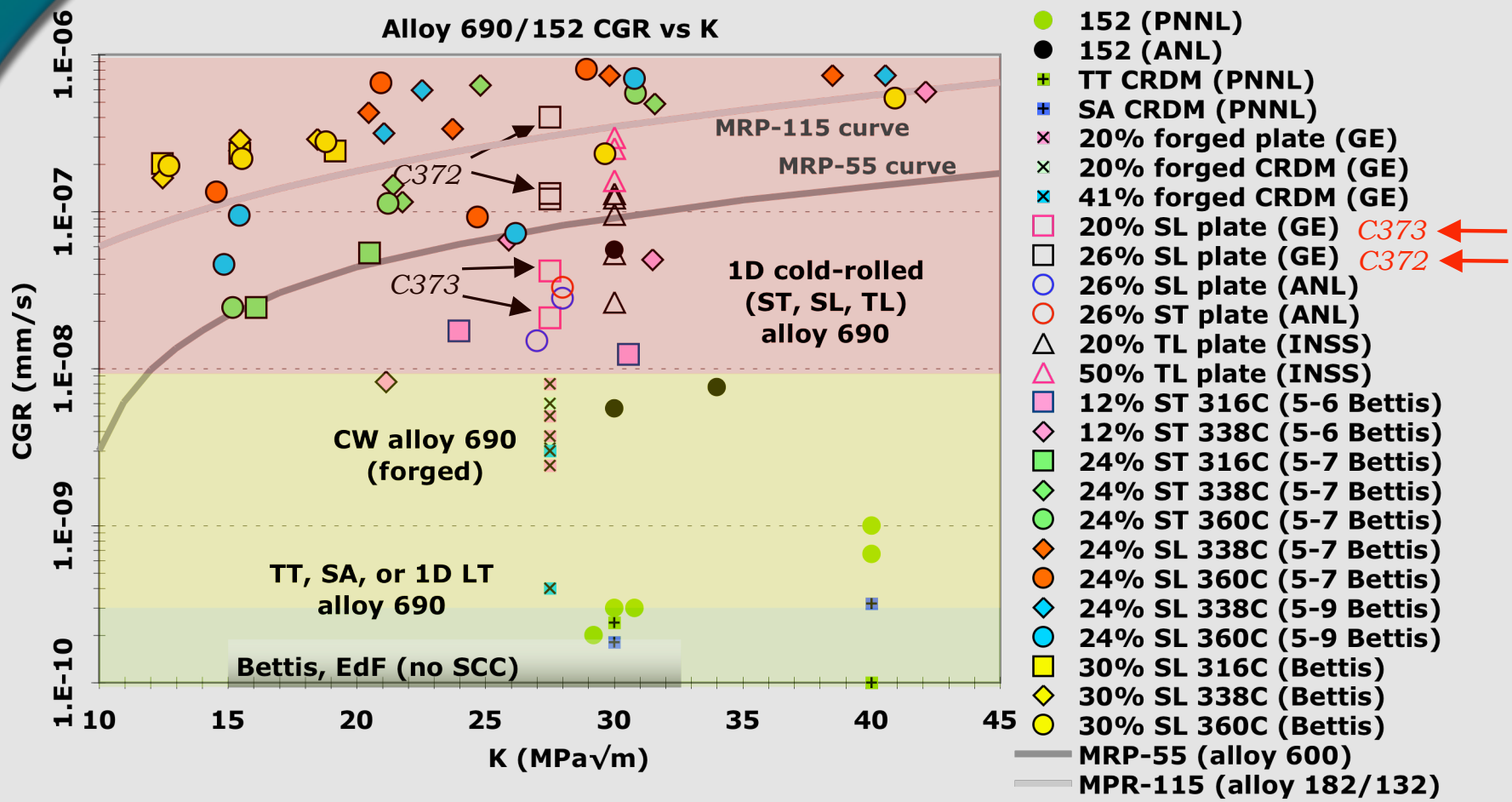
Alloy 690/152 Data (Excluding 1D Rolled)



Limited CGR data for LWR material conditions of alloy 690/152/52. Many tests report no SCC.

- *As-received, thermally treated or solution annealed alloy 690 exhibits extremely low constant K, CGRs (<5x10^{-10} mm/s) or no SCC. Cold work by forging increases CGRs, still <1x10^{-8} mm/s.*
- *Alloy 152 weld metal has shown variable behavior.*

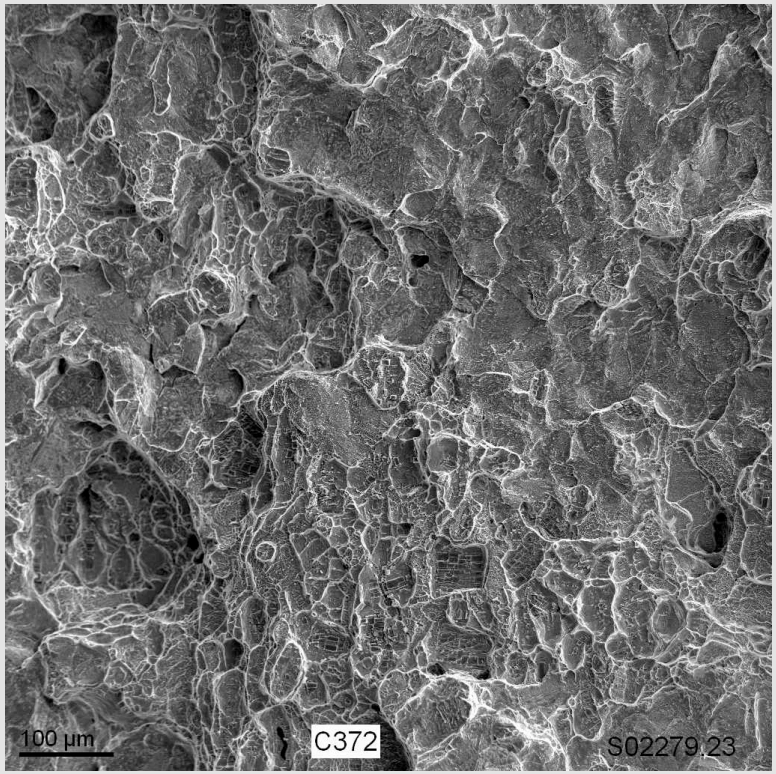
All Alloy 690/152 Data - K Dependence

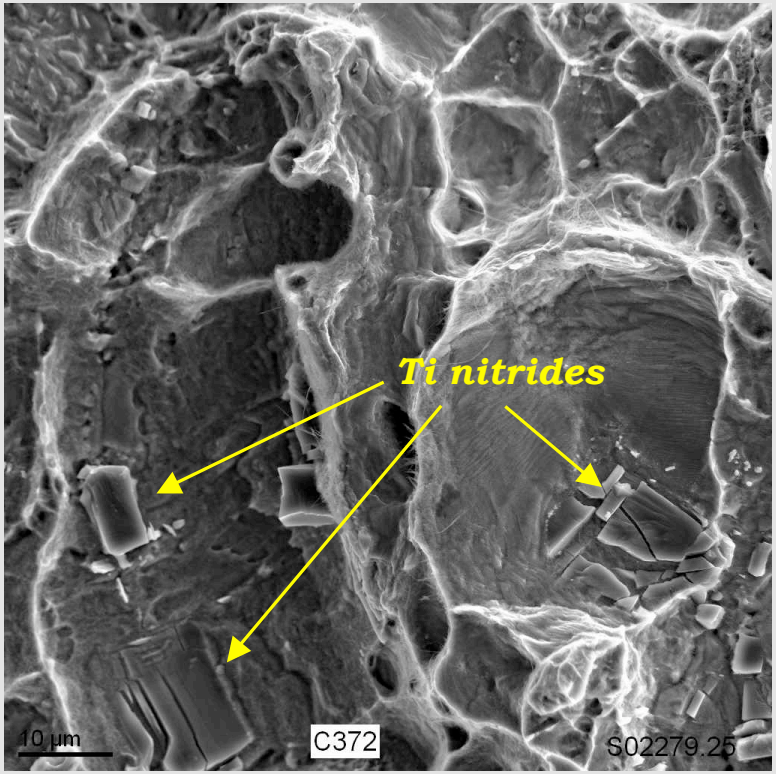


- *1D cold-rolled (S-L, S-T, T-L) materials exhibit high CGRs at constant K or constant load even at lower K values.*
- *Are 1D rolled materials relevant for LWR service components? Must understand high SCC susceptibility.*

Alloy 690 Plate Heat NX3297HK12 ANL 26% Cold Rolled

Material processed at ANL, CGR tests at ANL and GEG
GEG Test Sample C372 supplied by Peter Andresen

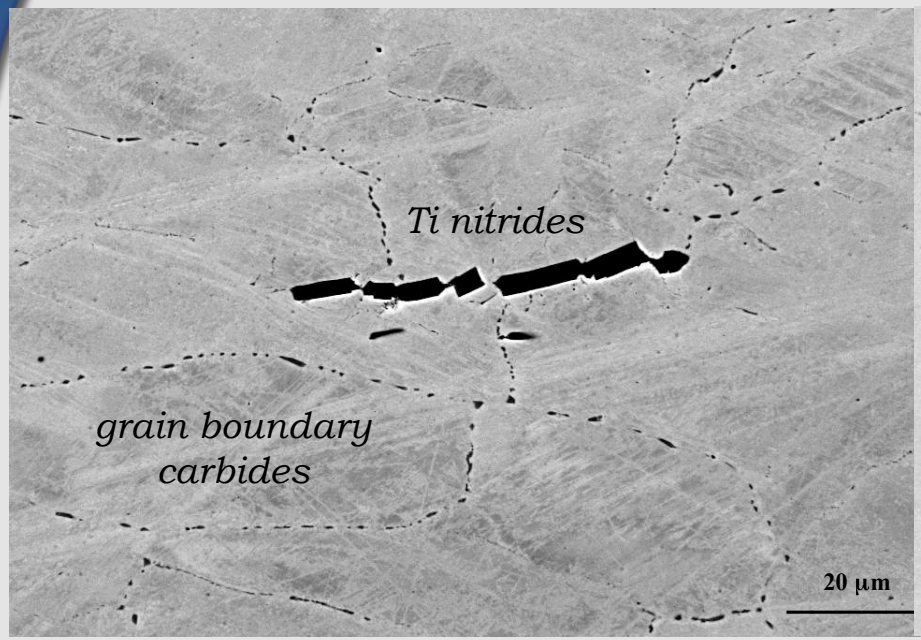




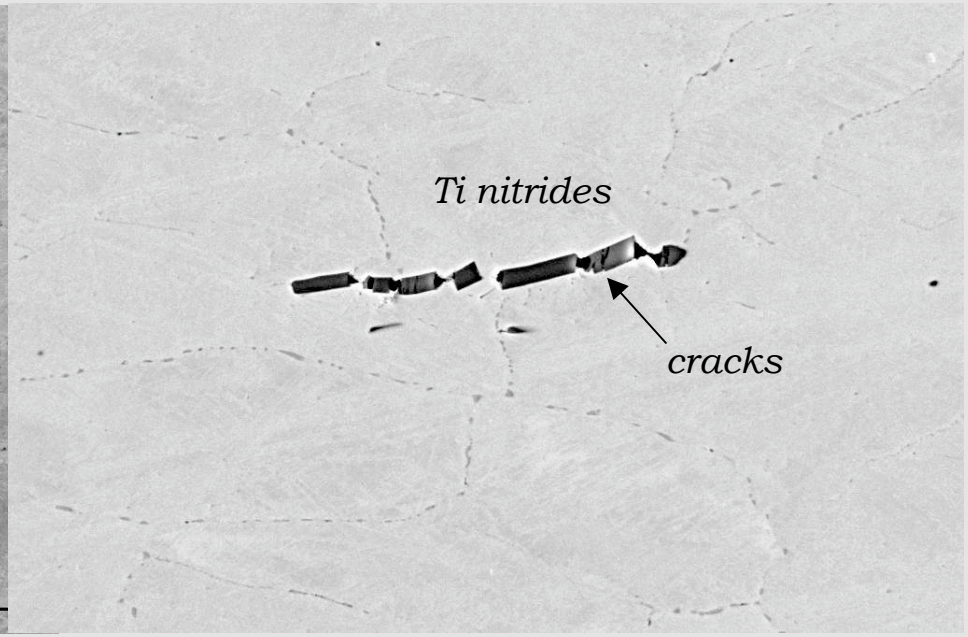
Mixed-mode fracture surface with IG (described as granulated) and TG regions, tearing regions often contain cracked Ti nitrides.

Alloy 690 Plate Heat NX3297HK12 ANL 26% Cold Rolled

GEG Test Sample C372



SEM-BSE



SEM-SE

RD →

Metallographic examination of area far from fracture surface. Evidence for grain elongation along the rolling direction. High density of fine grain boundary $Cr_{23}C_6$ carbides with moderate density of large, cracked Ti nitrides.

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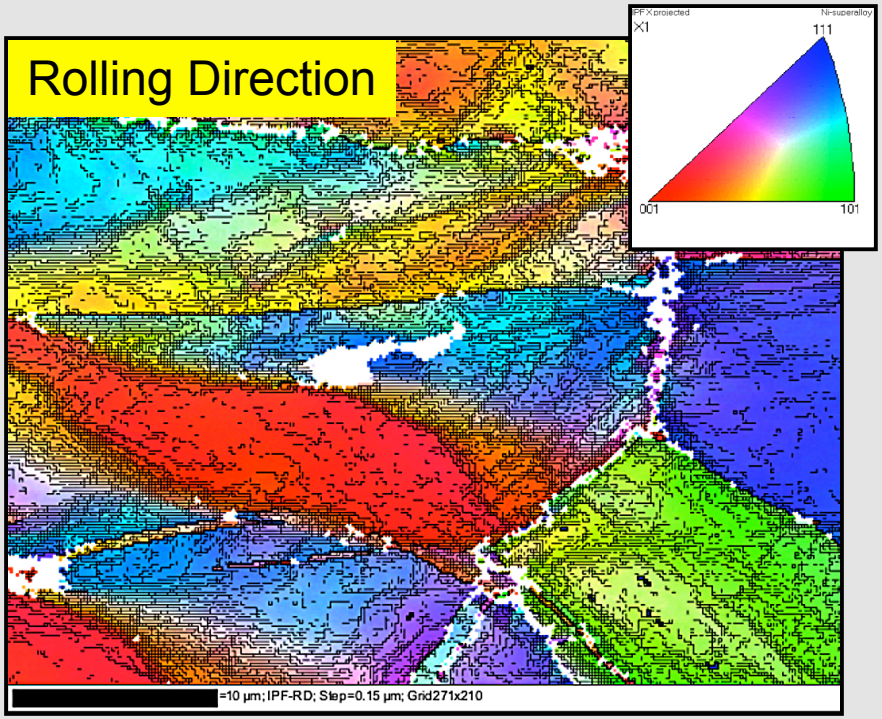

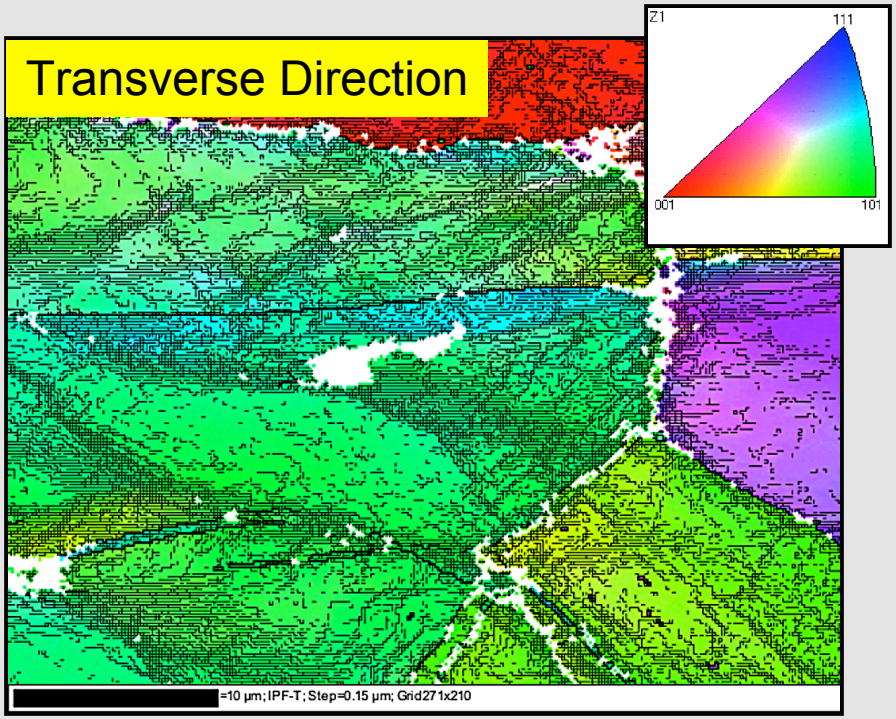

Comparison of inverse pole figure maps (RD versus TD): RD orientation map shows a greater degree of lattice misorientation than in the TD map. Boundaries are shown with regions of >0.75° misorientation. Difficulty indexing near matrix TiN or grain boundary $M_{23}C_6$ particles.

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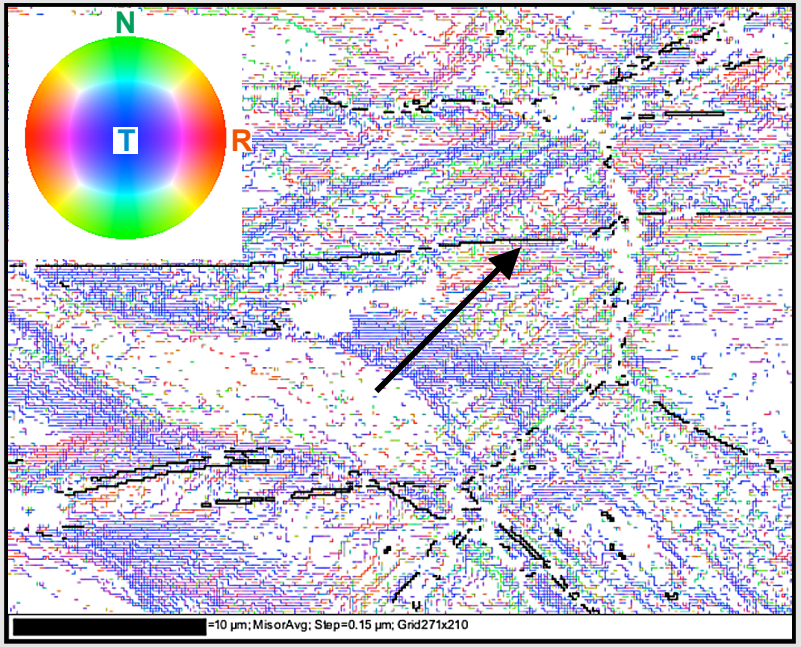
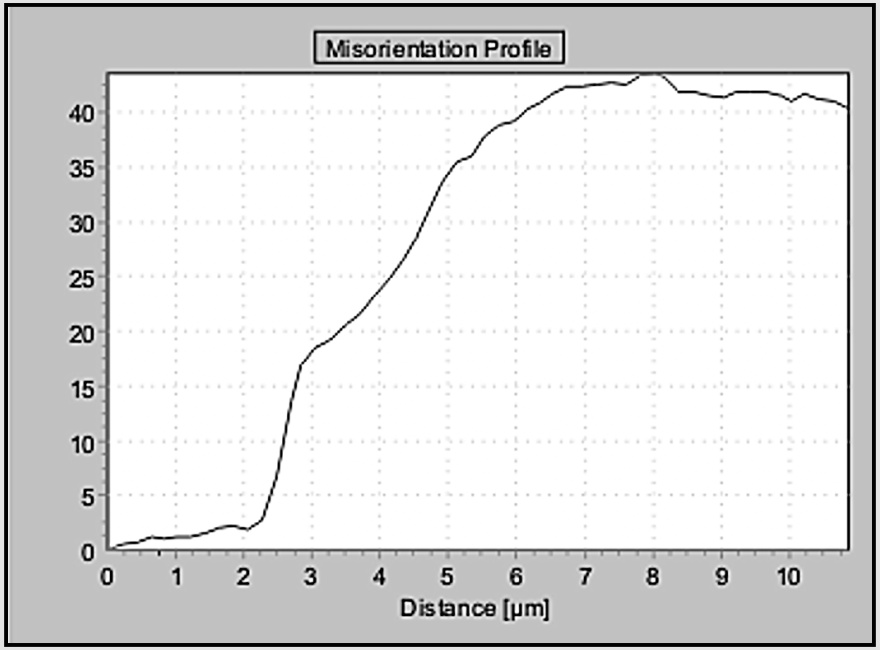
GEG Test Sample C372

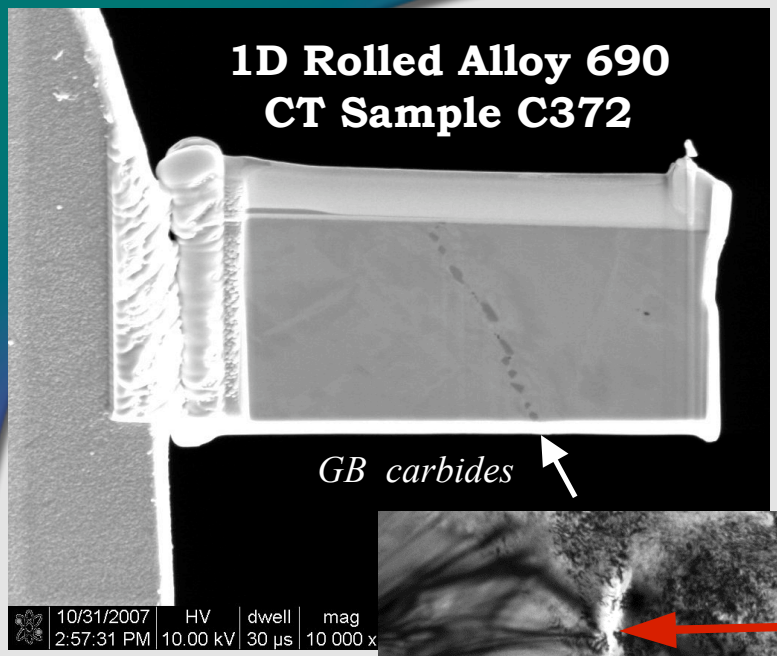


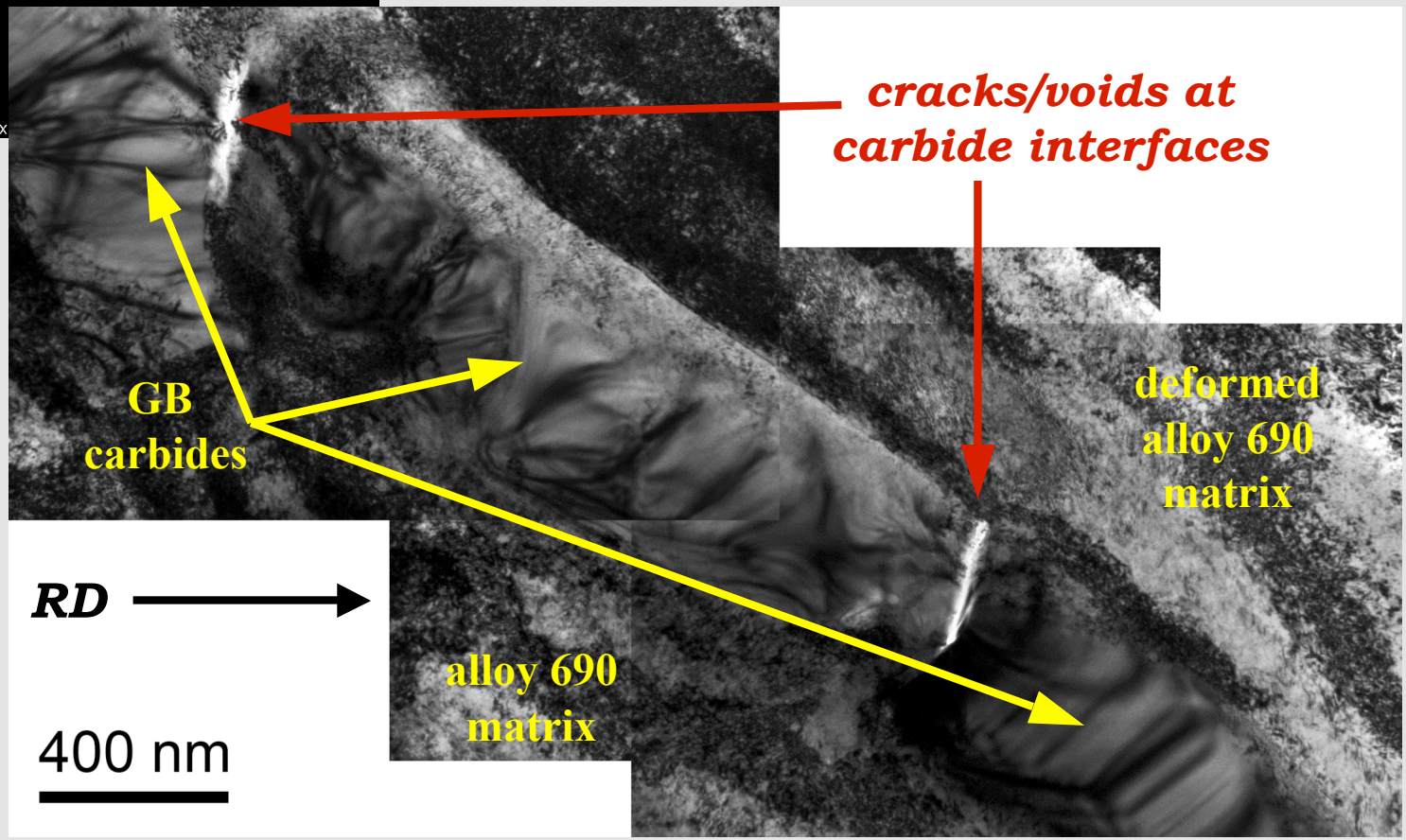


Misorientation axis map: Blue boundaries indicate the axis of rotation is the transverse (T) direction, red boundaries are rotated about the rolling (R) direction. Misorientation profile near TiN particle in the middle region (black arrow) shows the degree of misorientation across the grain to the boundary.

TEM Cross-Section Sample Preparation Using a Focused-Ion Beam (FIB) System

FIB is being used to evaluate nm-scale damage along grain boundaries in 1D-rolled alloy 690 that show high IGSCC crack-growth rates during tests in PWR primary water. ***Nanoscale cracks and/or voids*** *found by TEM at grain boundary carbide interfaces.*





FIB has removed samples from regions far from the CT fracture surface. Samples cut from several different section orientations in an attempt to assess the 3D grain boundary characteristics.

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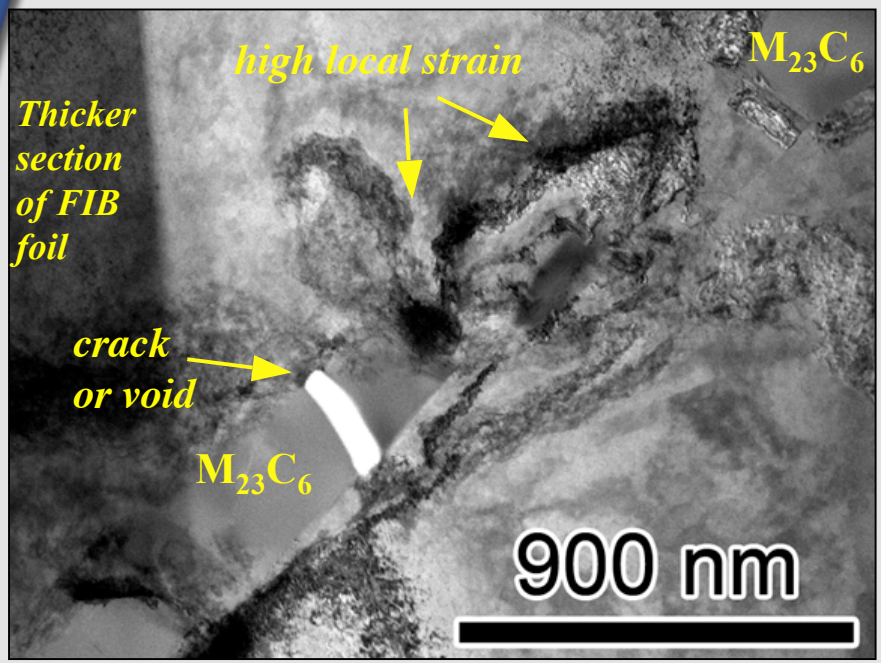
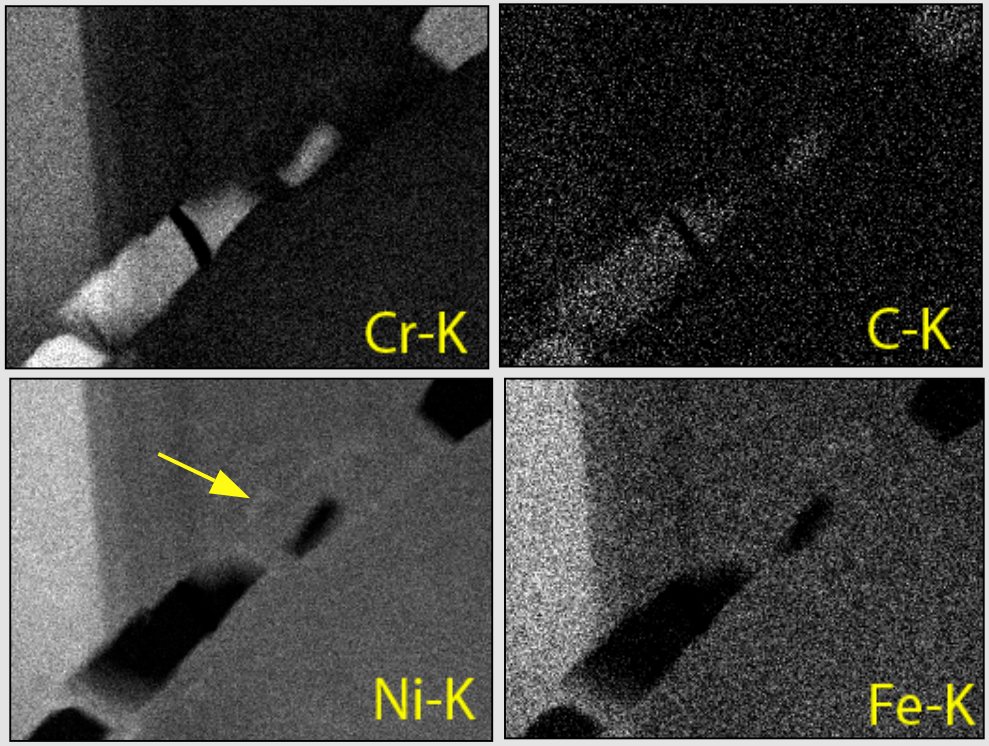
Alloy 690 Plate Heat NX3297HK12 ANL 26% Cold Rolled

GEG Test Sample C372



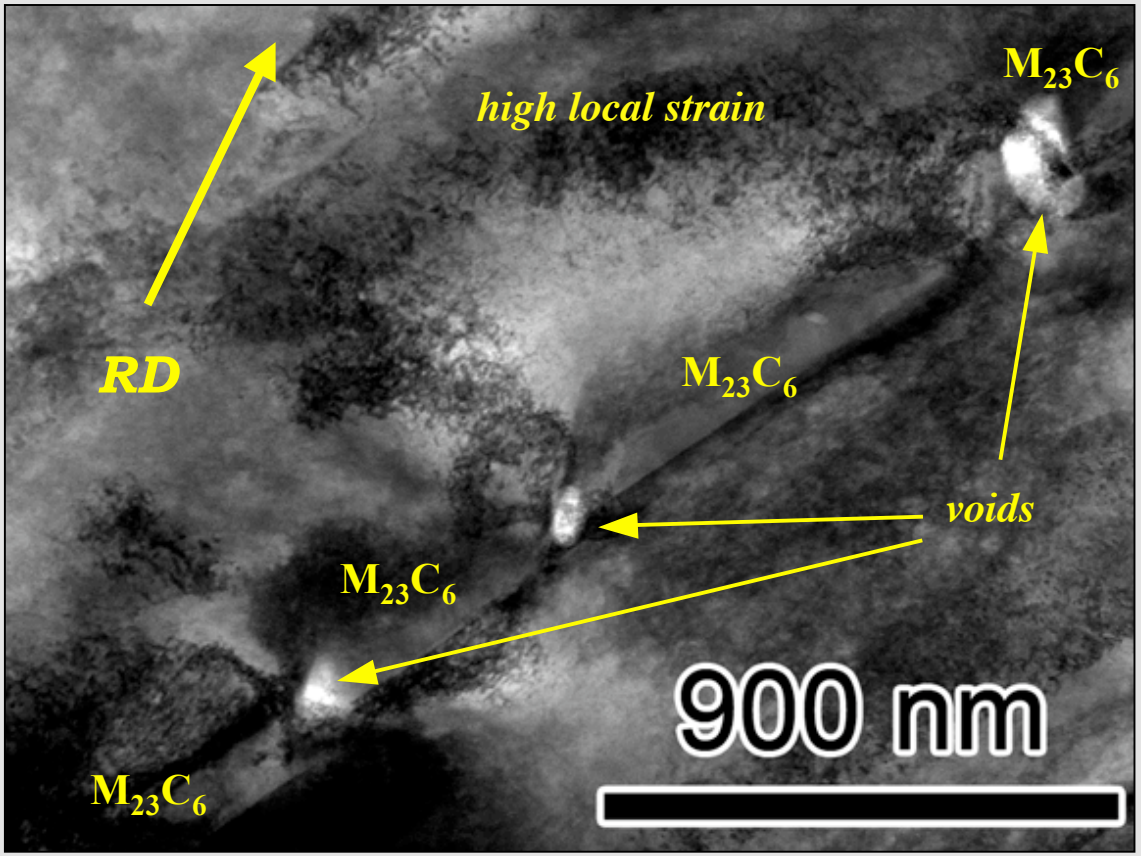
RD →



TEM image from third series of FIB samples; crack separating carbides, high local strain at all carbide interfaces. EDS maps illustrating GB Cr carbides and Cr depletion.

Alloy 690 Plate Heat NX3297HK12 ANL 26% Cold Rolled

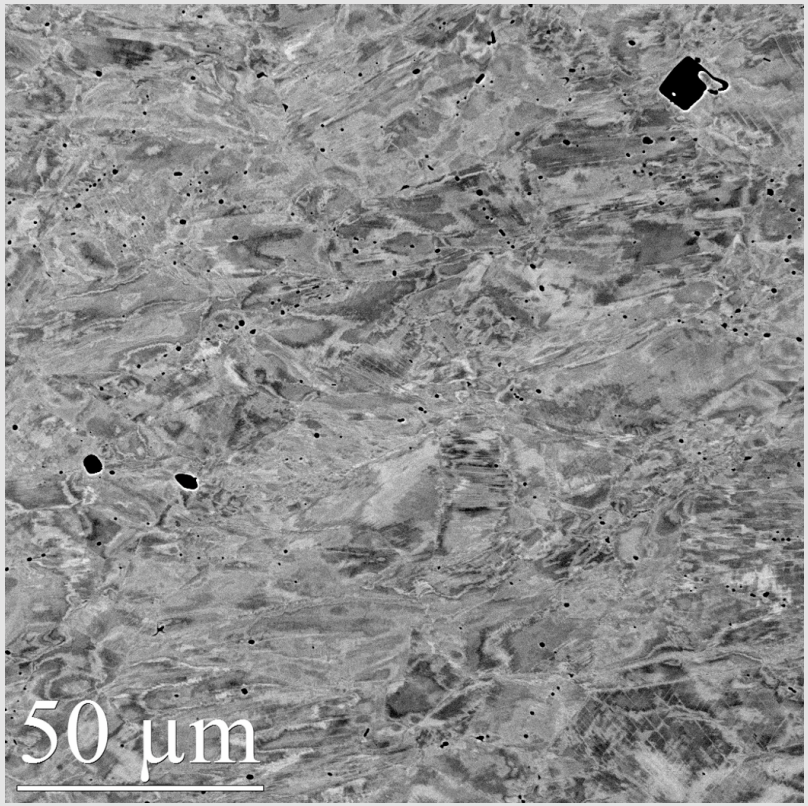
GEG Test Sample C372

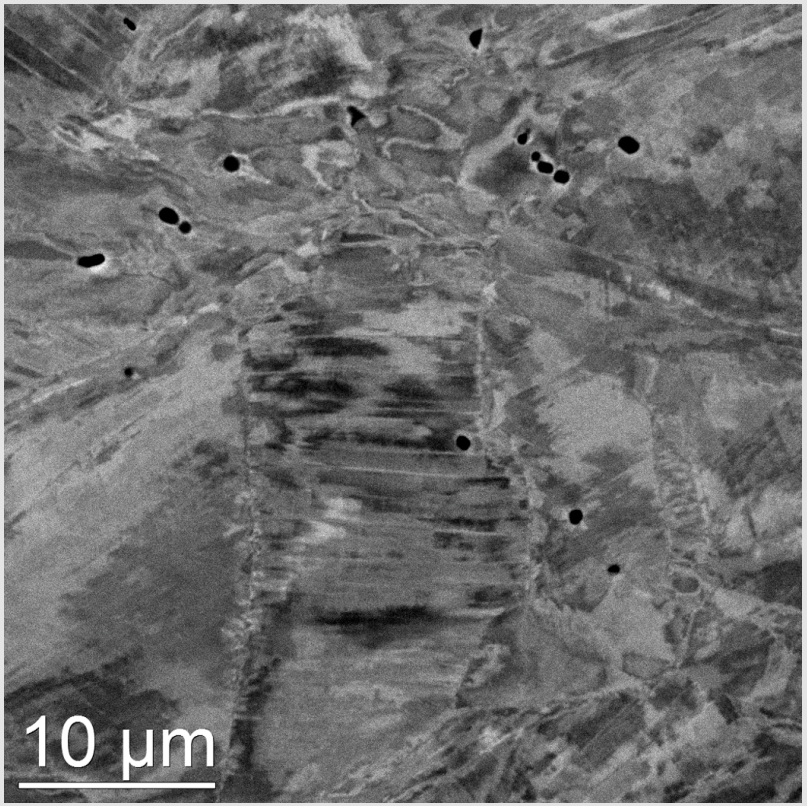


TEM image from third series of FIB samples (two orientations); voids separating carbides, high local strain at voids and at carbide interfaces.

Alloy 690 Plate Heat B25K GE Global 20% 1D Cold Rolled

GEG Test Sample C373 supplied by Peter Andresen



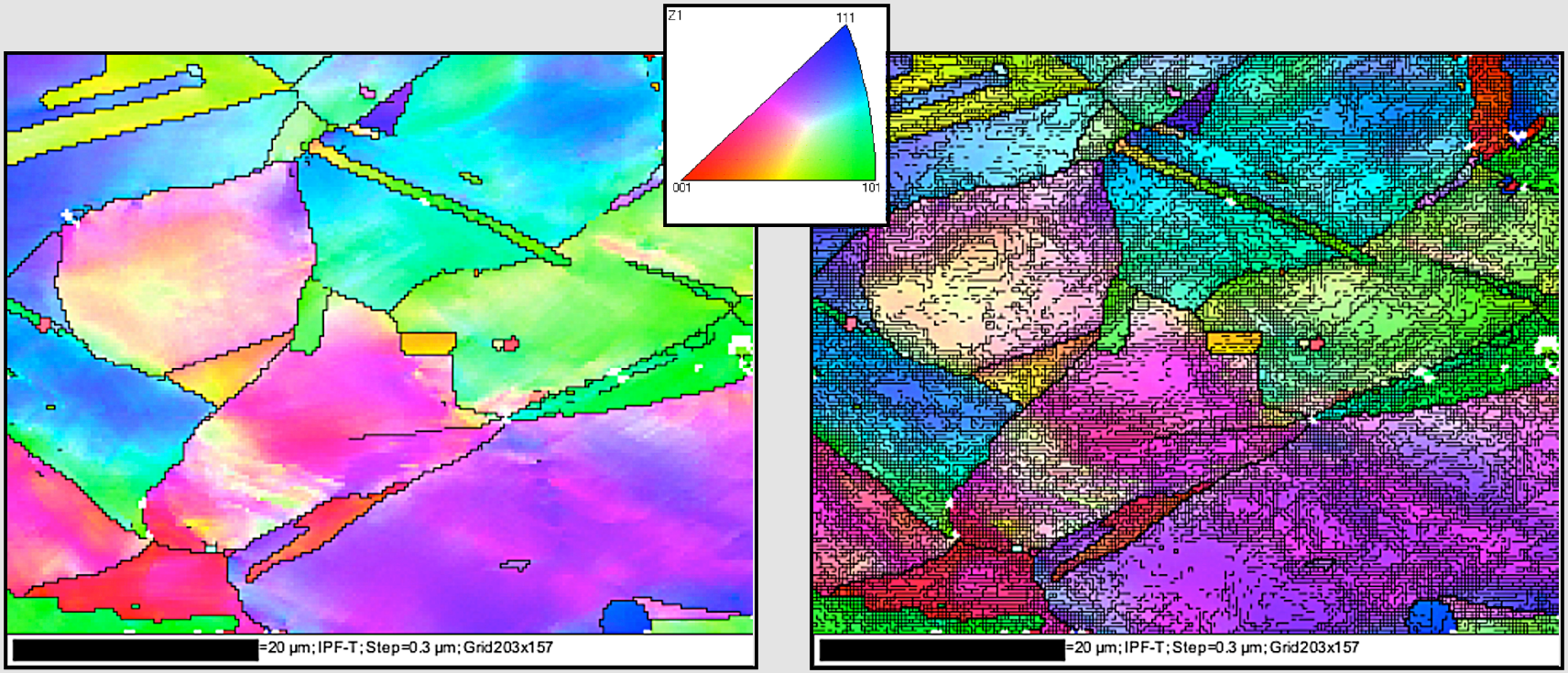


RD →

BSE-SEM images show a highly deformed grain structure with few visible carbides on the grain boundaries, but moderate density of large TiNi particles.

Alloy 690 Plate Heat B25K GEG 20% 1D Cold Rolled

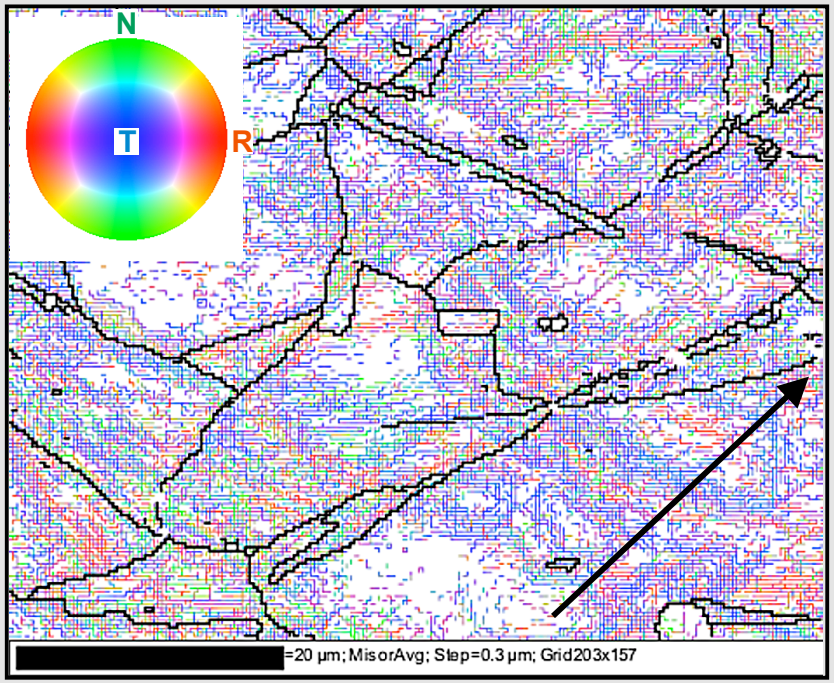
GEG Test Sample C373

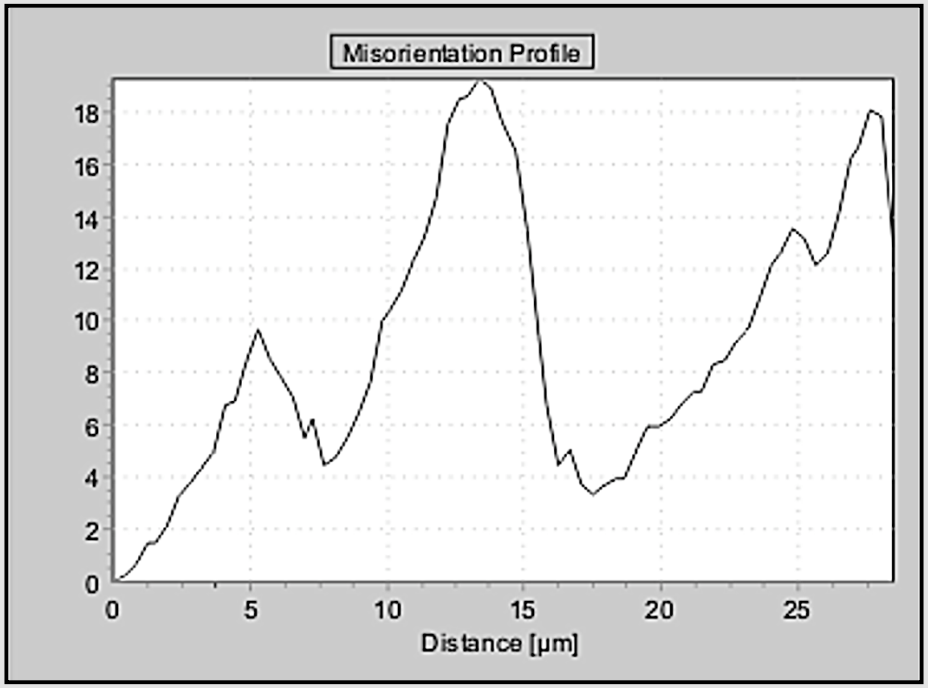


Inverse pole figure map (transverse direction): Non-uniform colors indicate lattice rotation across highly strained grains. Map shows boundaries with regions of greater than >0.75° misorientation.

Alloy 690 Plate Heat B25K GEG 20% 1D Cold Rolled

GEG Test Sample C373

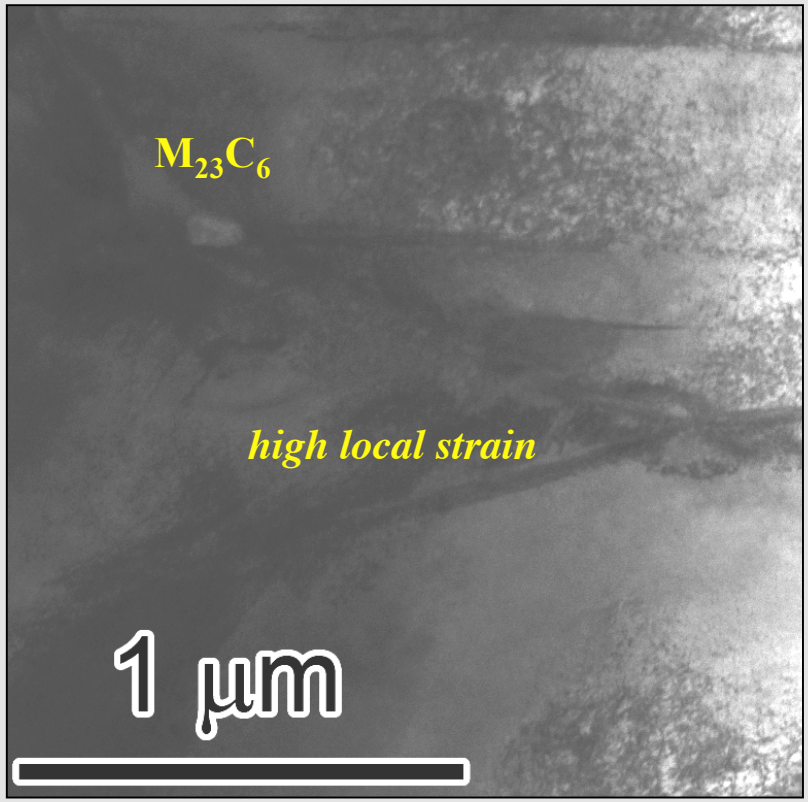


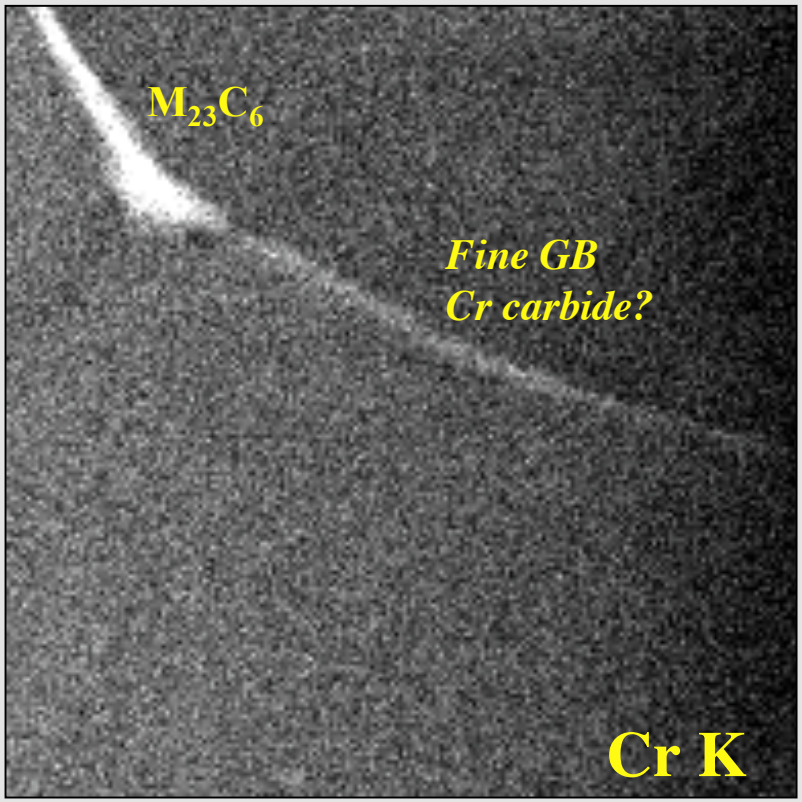


Misorientation axis map: Blue boundaries indicate the axis of rotation is the transverse (T) direction, red boundaries are rotated about the rolling (R) direction. Misorientation profile across grain in lower left hand region (black arrow) shows the degree of misorientation across the grain to the boundary.

Alloy 690 Plate Heat B25K GEG 20% 1D Cold Rolled

GEG Test Sample C373
Very limited observations (FIB samples too thick)





TEM images from FIB-TEM sample showing elongated grain boundary $M_{23}C_6$ carbide and Cr enrichment along GB in EDS map; high local strain associated with grain boundaries and carbide interfaces, but no observations of cracks/voids at interfaces.

1D Cold Rolled Alloy 690

- *1D cold rolling to 26% reduction (C372) produced:*
  - *grain elongation in rolling plane*
  - *extensive cracking of matrix Ti-nitride particles with high local plastic strains; cracks/voids and high local plastic strains at grain boundaries and intergranular carbides*
  - *Rolling-induced damage can create a pre-cracked microstructure, however far from a continuous path and mechanism remains unclear to explain the ~100X increase in SCC propagation rates.*
- *Limited results on GEG 20% cold rolled C373 show similar strain distributions, better FIB-TEM needed to assess boundary damage structures.*
- *Micro-to-nanoscale examinations continuing on four 1D cold rolled and two forged alloy 690 materials to comparable levels of cold work.*