INL/CON-21-62704-Revision-0



# Effect of Water Chemistry on Crack Growth Rate in Neutron Irradiated X-750 and XM-19

July 2022

Drew Coulson Johnson, John Howard Jackson, Michael P Heighes, Robert G. Carter, Peter L. Andresen

Changing the World's Energy Future

Idaho National Laboratory

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**July 2022**

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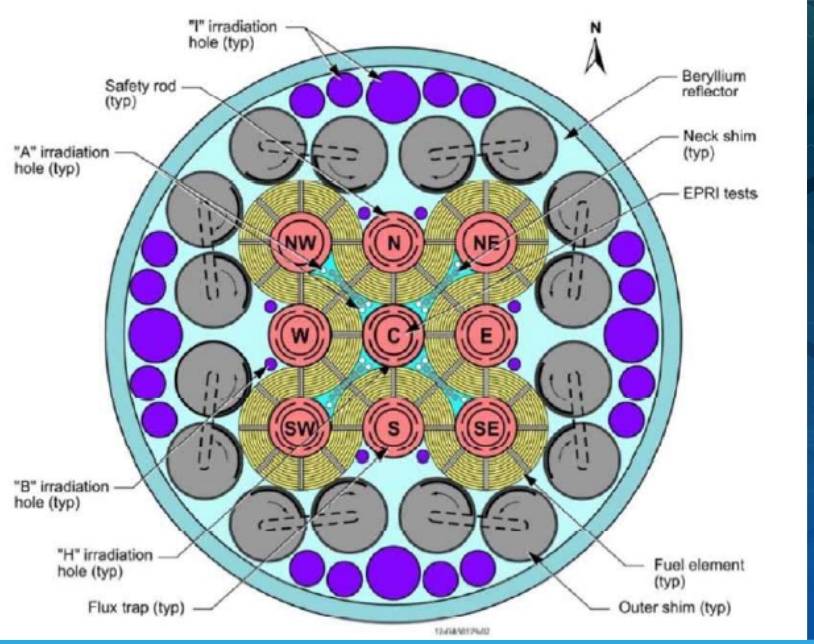
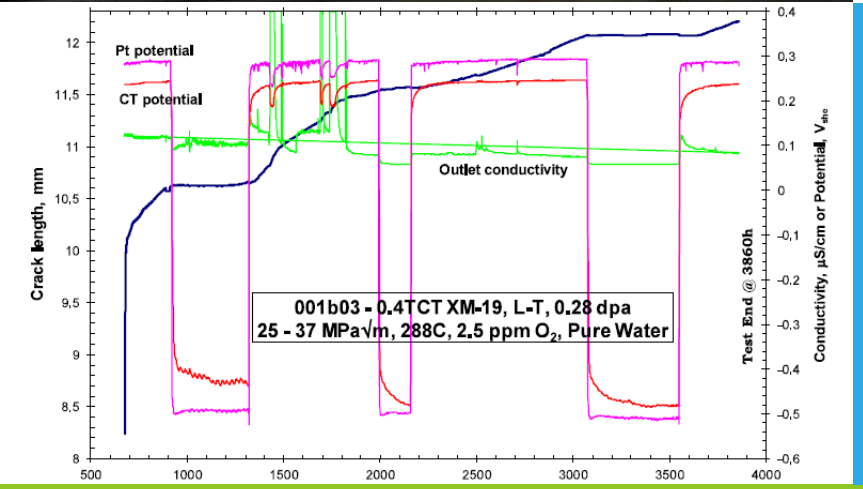




Drew C. Johnson

ENV DEG 2022

Tuesday July 19th


# Effect of Water Chemistry on Crack Growth Rate in Neutron Irradiated X-750 and XM-19

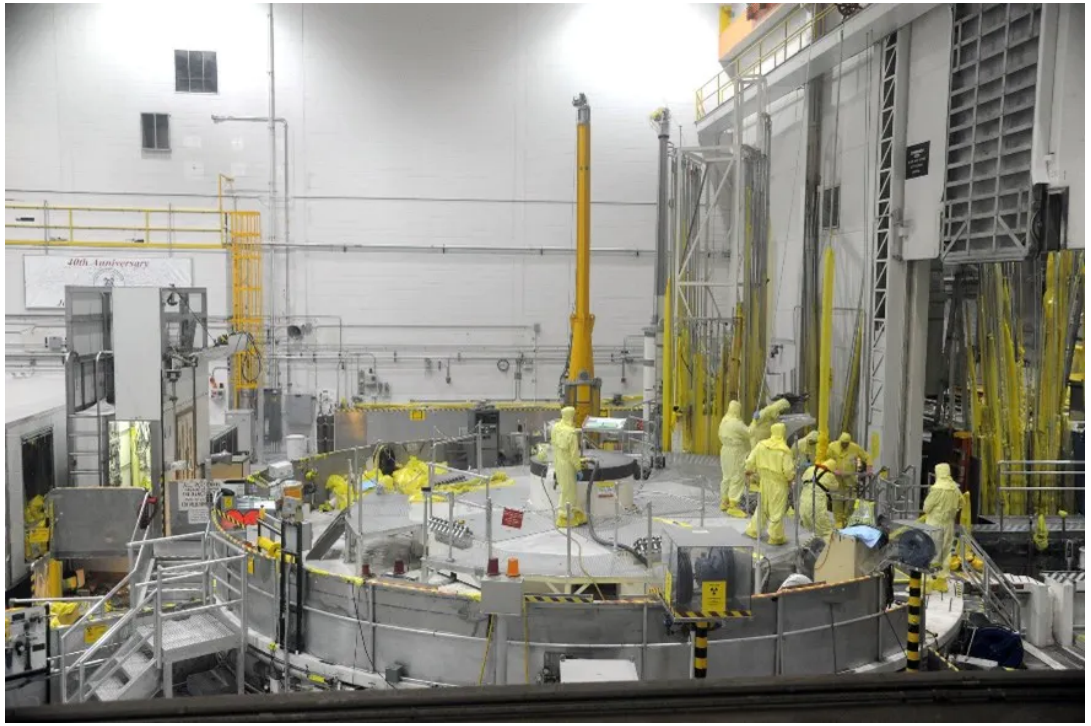

Idaho National Laboratory

# Co-authors

- John H. Jackson and Michael Heighes, INL
- Peter L. Andresen, Andresen Consulting/GE Global Research Center (retired)
- Robert Carter, EPRI

# Neutron Irradiation and PIE of X-750 and XM-19

- Irradiations conducted at the Advanced Test Reactor (ATR) National Science User Facility (NSUF), utilizing the central flux trap (CFT) position
  - First civilian project to utilize this reactor position
- ~0.3 dpa and ~1.4 dpa samples produced to investigate mid-life and life extension (60-80 yr) behavior
- 0.4T-CT specimens used for crack growth rate
- Effect of water electro-chemical potential and conductivity evaluated for both alloys at each irradiation condition



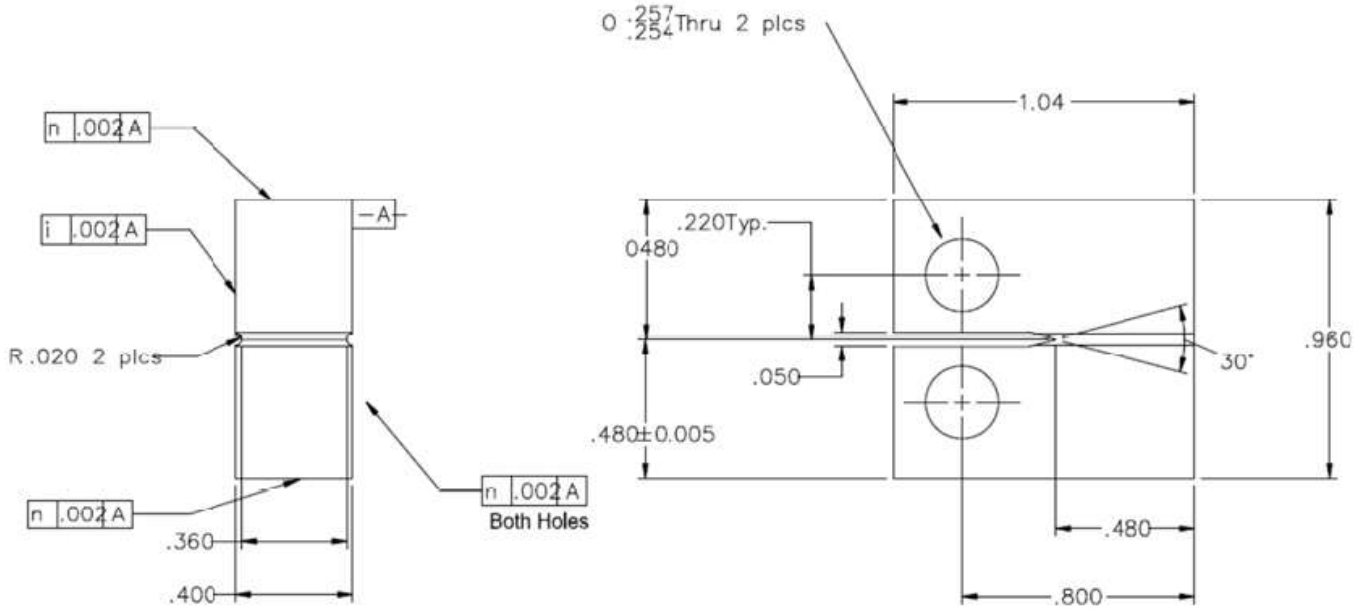
# X-750 and XM-19 Samples for CGR Testing

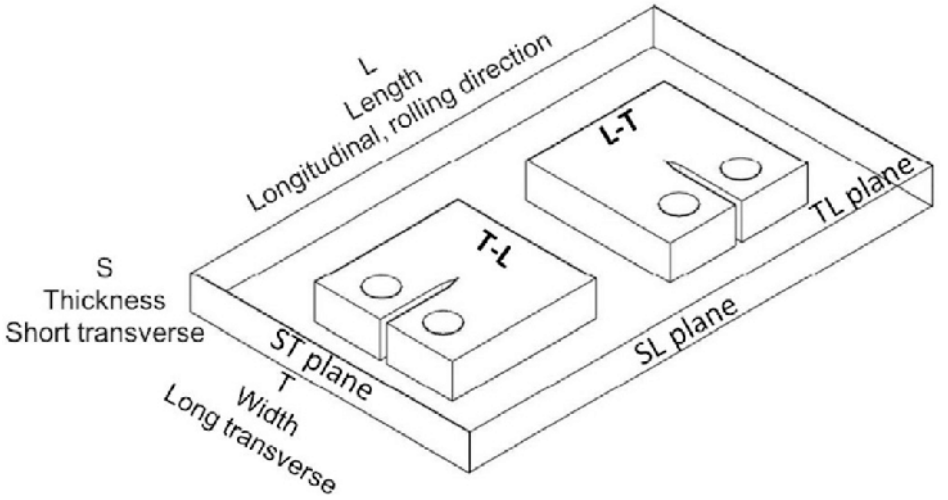
## X-750

| Element | Al | C | Nb | Co | Cr | Cu | Fe | Mn | Ni | S | Si | Ti | P | Ta |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| wt % | 0.77 | 0.04 | 0.97 | 0.726 | 14.99 | 0.015 | 7.8 | 0.197 | 70.8 | 0.002 | 0.25 | 2.42 | <0.005 | <0.01 |

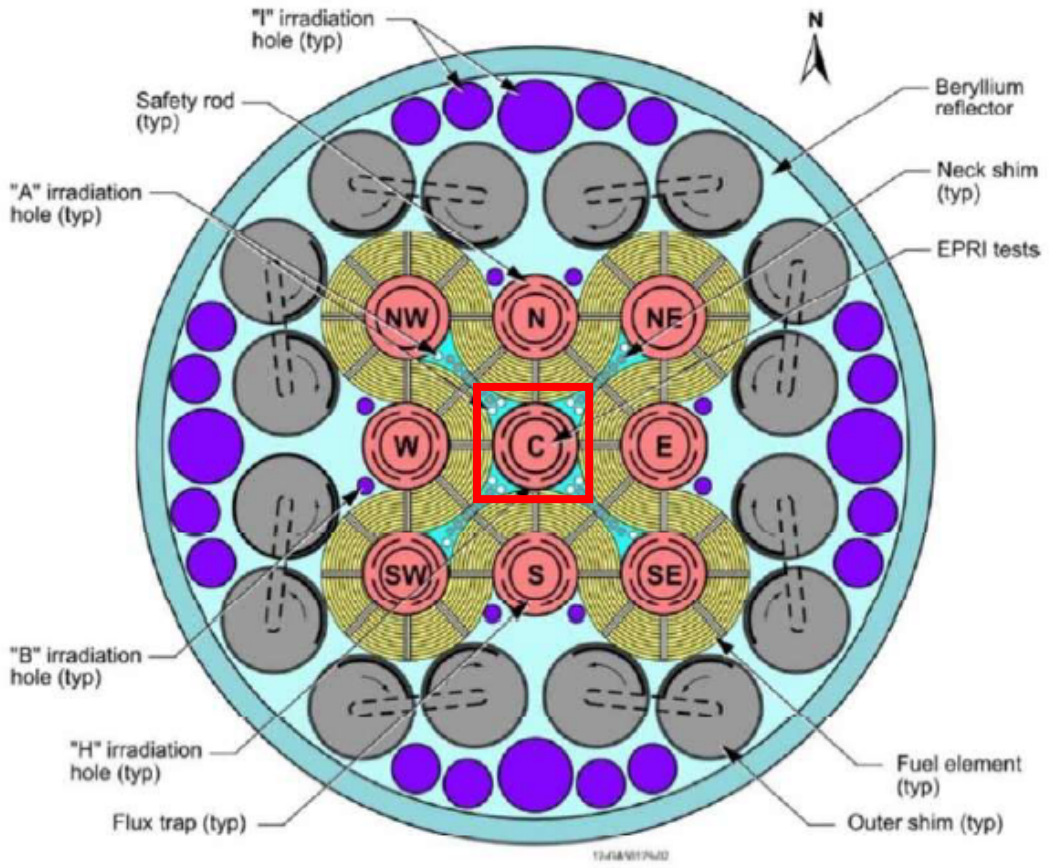
## XM-19

| Element | Mo | C | Nb | Co | Cr | Cu | Fe | Mn | Ni | S | Si | Ti | P | W |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| wt % | 2.17 | 0.027 | 0.2 | 0.016 | 20.76 | 0.06 | 58.88 | 4.59 | 12.82 | 0.06 | 0.39 | 0.001 | 0.014 | 0.02 |

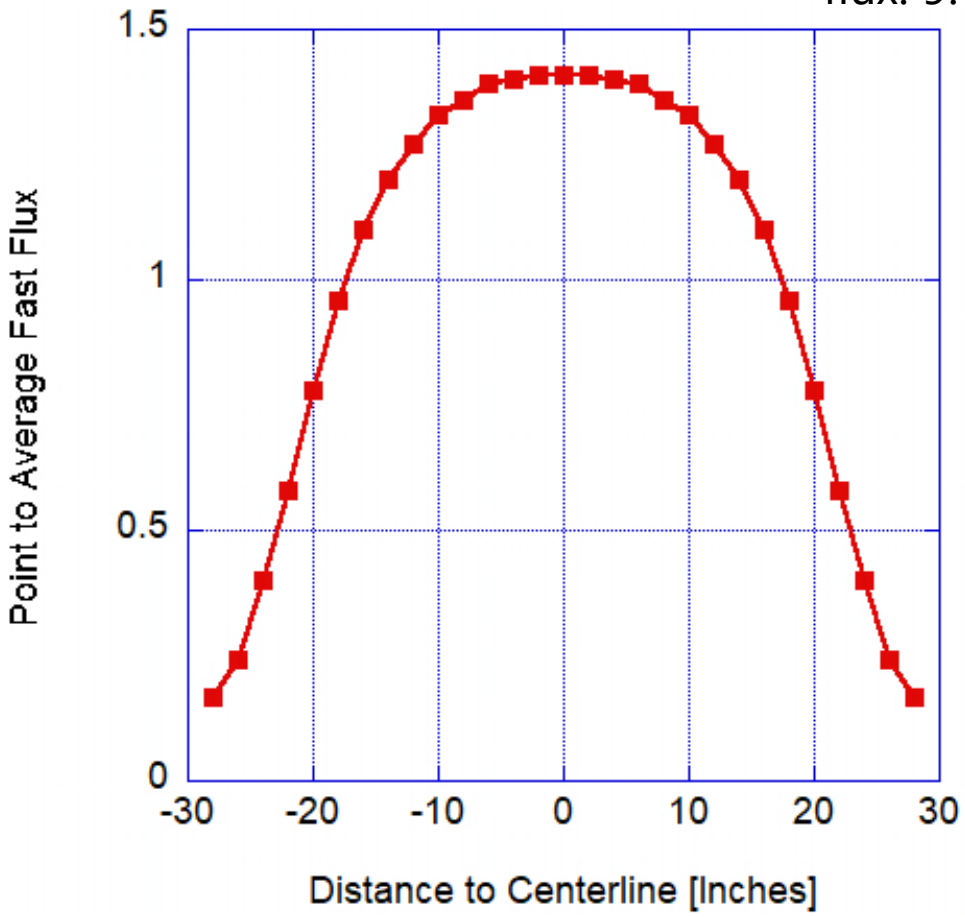


# ATR Testing Location and Normal Operating Flux




ATR CFT nominal fast flux: 9.7 x $10^{13}$ n/$cm^2$-s



| Position | Cycle - 1 | Cycle - 2 | Cycle - 3 | Cycle - 4 | Cycle - 5 | Cycle - 6 | Cycle - 7 | Cycle - 8 | Cycle - 9 | Cycle - 10 | Cycle - 11 | Cycle - 12 | Cycle - 13 | Cycle - 14 | Cycle - 15 | Cycle - 16 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Position 1 | N-1 | N-2 | N-2 | N-5 | N-5 | N-5 | N-5 | N-5 | N-5 | 316H-2 | 316H-2 | 316H-2 | 800H-1 | 709-2 | 709-2 | 709-2 |
| Position 2 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-20 | N-10 | N-10 | N-10 | N-10 | N-10 |
| Position 3 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | 709-20 | N-10 | 709-10 | 709-10 | 709-10 | 709-10 | 709-10 |
| Position 4 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-20 | 316H-10 | 316H-10 | 316H-10 | 316H-10 | 316H-10 |
| Position 5 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-20 | 800H-2 | 800H-10 | 800H-10 | 800H-10 | 800H-10 | 800H-10 |
| Position 6 | 316H-1 | 709-1 | 800H-5 | 800H-5 | 800H-5 | 800H-5 | 709-5 | 709-5 | 709-5 | 709-5 | 316H-5 | 316H-5 | 316H-5 | 316H-5 | 316H-5 | BLANK |

# Measured Parameters for ATR Irradiation

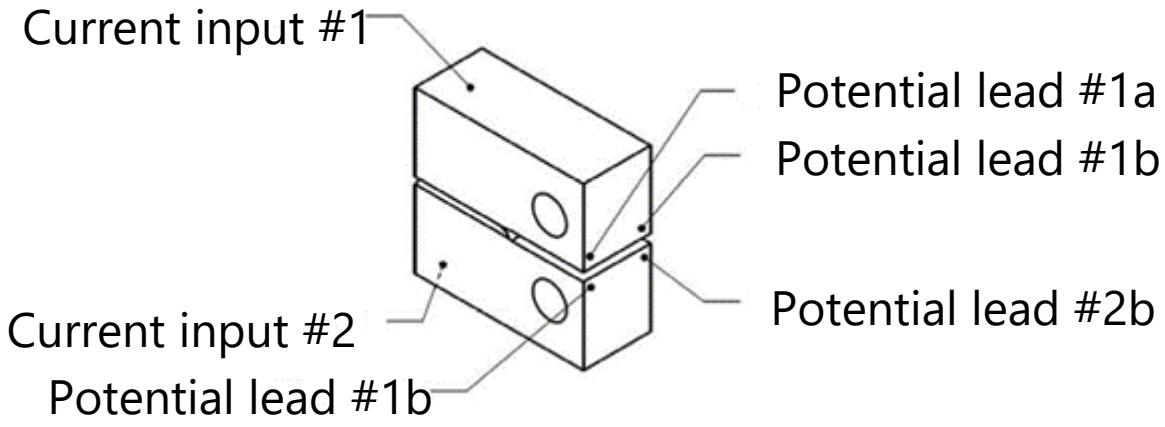
- MCNP analysis used to perform estimate of accumulated fluence
- ABAQUS model compared to packages of melt wires to estimate sample temp during irradiation
- Multiple 0.4T-CT samples of each alloy were tested from the ~0.3 and ~1.4 dpa irradiations

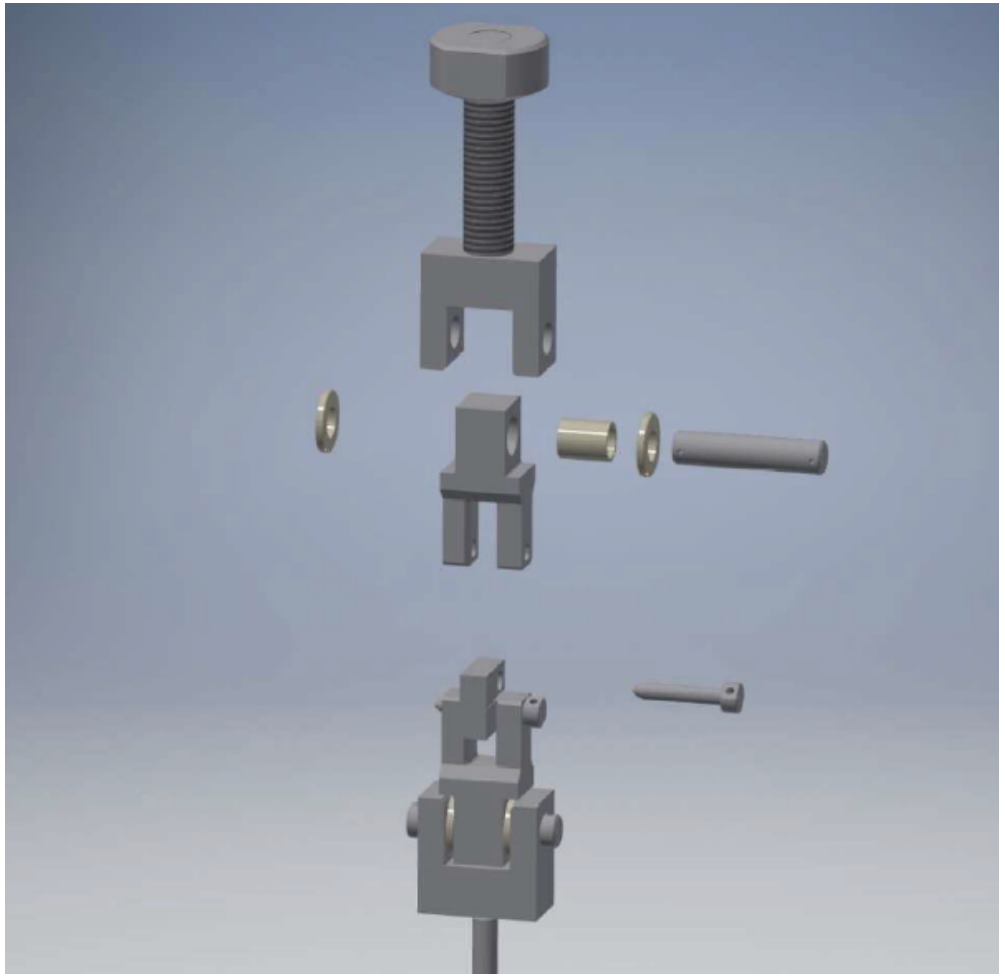
| Specimen ID | Alloy | Fast Fluence (x$10^{20}$) | dpa | Min Temp [°C] | Max Temp [°C] |
|---|---|---|---|---|---|
| 10A0002 A10 | X-750 | 1.93 | 0.309 | 349 | 359 |
| 10A0002 A08 | X-750 | 1.89 | 0.302 | 329 | 338 |
| 10A0001 B03 | XM-19 | 1.93 | 0.294 | 349 | 359 |
| 10A0001 B02 | XM-19 | 1.89 | 0.291 | 350 | 369 |
| 10A0002 B03 | X-750 | 8.658 | 1.441 | 287/257 | 295/259 |
| 10A0002 B09 | X-750 | 9.704 | 1.542 | 335/265 | 347/268 |
| 10A0001 D02 | XM-19 | 9.487 | 1.443 | 329/262 | 341/265 |
| 10A0001 B07 | XM-19 | 8.658 | 1.320 | 287/257 | 295/259 |

The higher fluence samples (bottom 4 rows in above table) were irradiated over 2 full cycles, resulting in temperature bounds for each cycle. Attempts to increase coolant flow through irradiation capsule redesign occurred between the irradiations cycles, resulting in significantly lower observed temperatures during the second cycle.

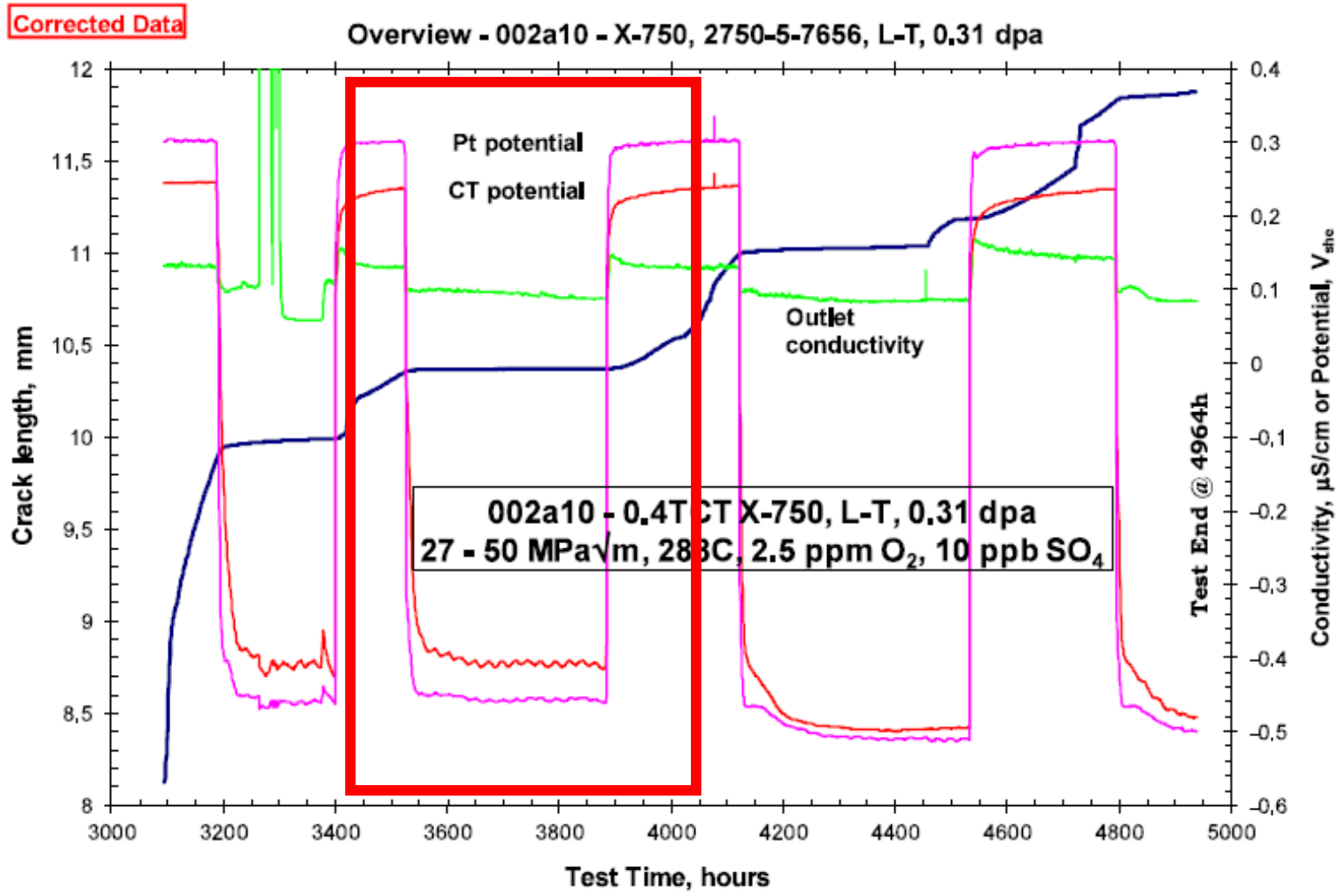
# CGR Testing of Irradiated Samples using DCPD

- 3 A direct current used for DCPD crack monitoring
- Water in autoclave during experiment kept at 288 °C, 1500 psig
- 10 ppb sulfate (as $H_2SO_4$) added to enhance reproducibility and allow for comparison with experiments using non-irradiated samples
- NWC: 2.5 ppm dissolved oxygen
- HWC: 60-90 ppb dissolved hydrogen

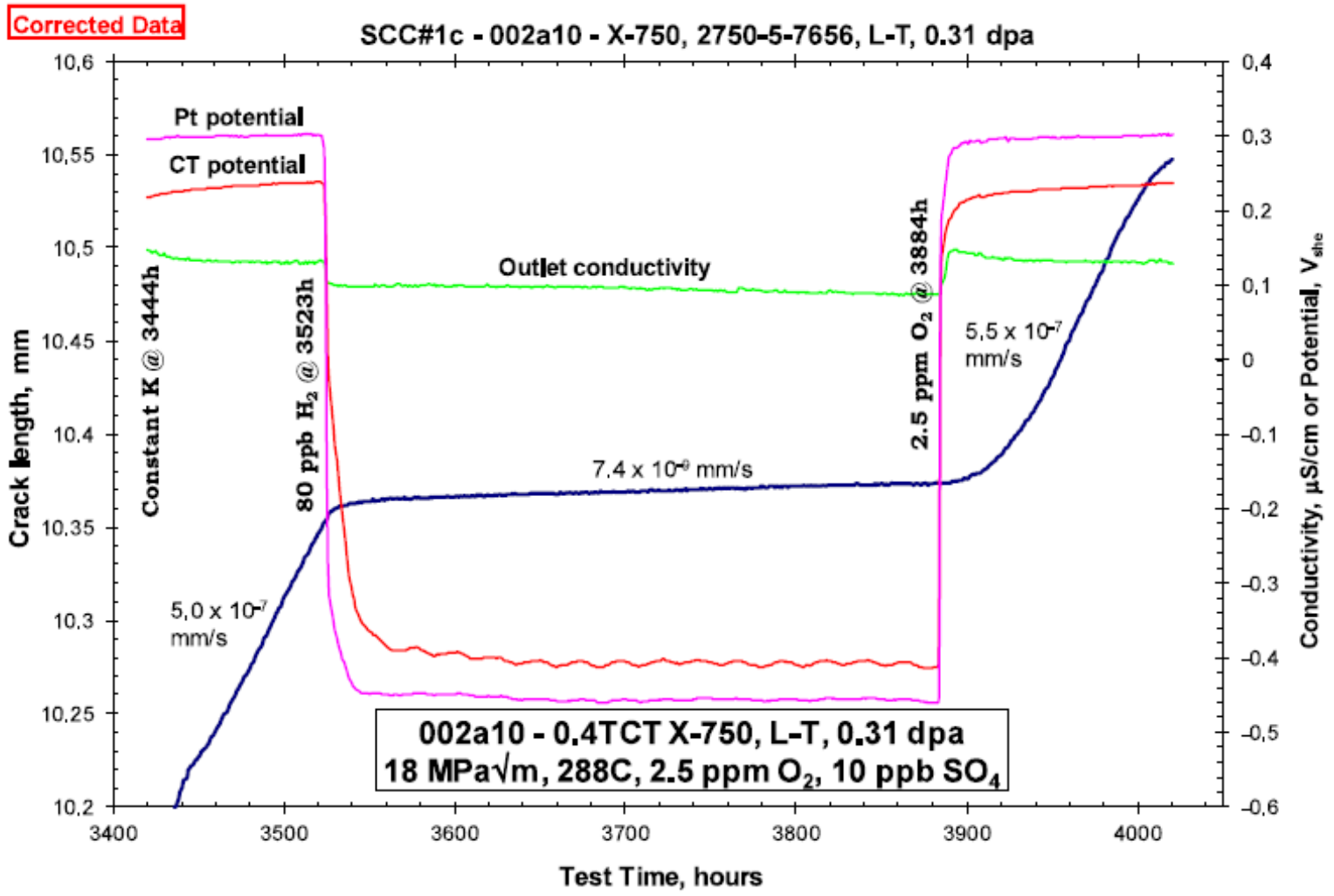


# X-750 ~0.3 dpa CGR (02A10) – Overview of CGR Test

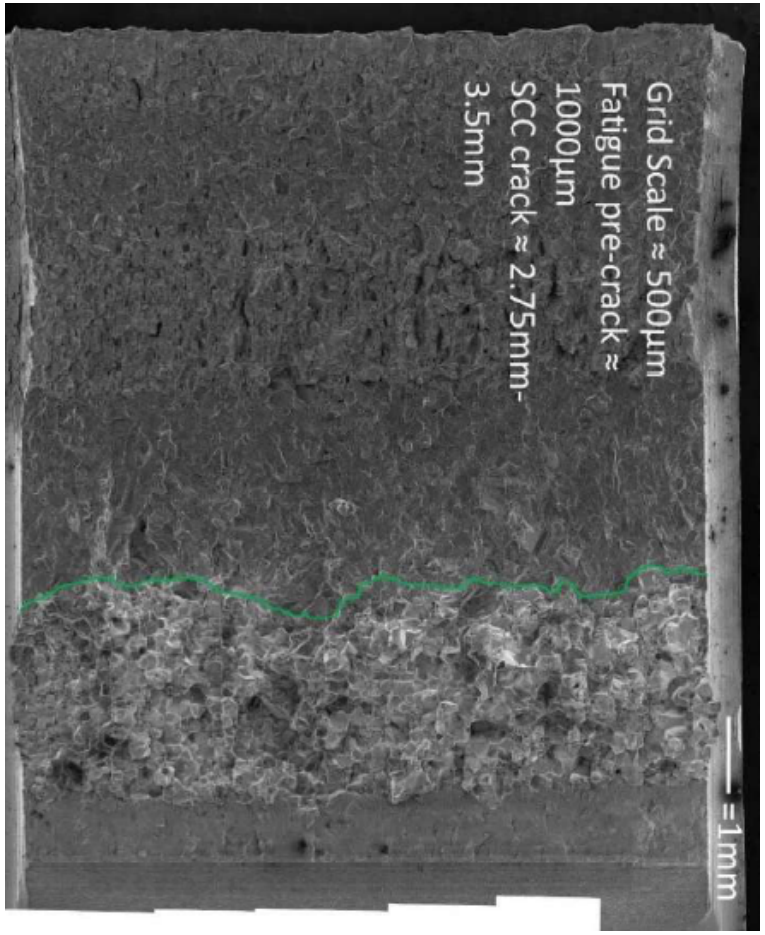


# X-750 ~0.3 dpa CGR (02A10) – Selected Evaluation Step



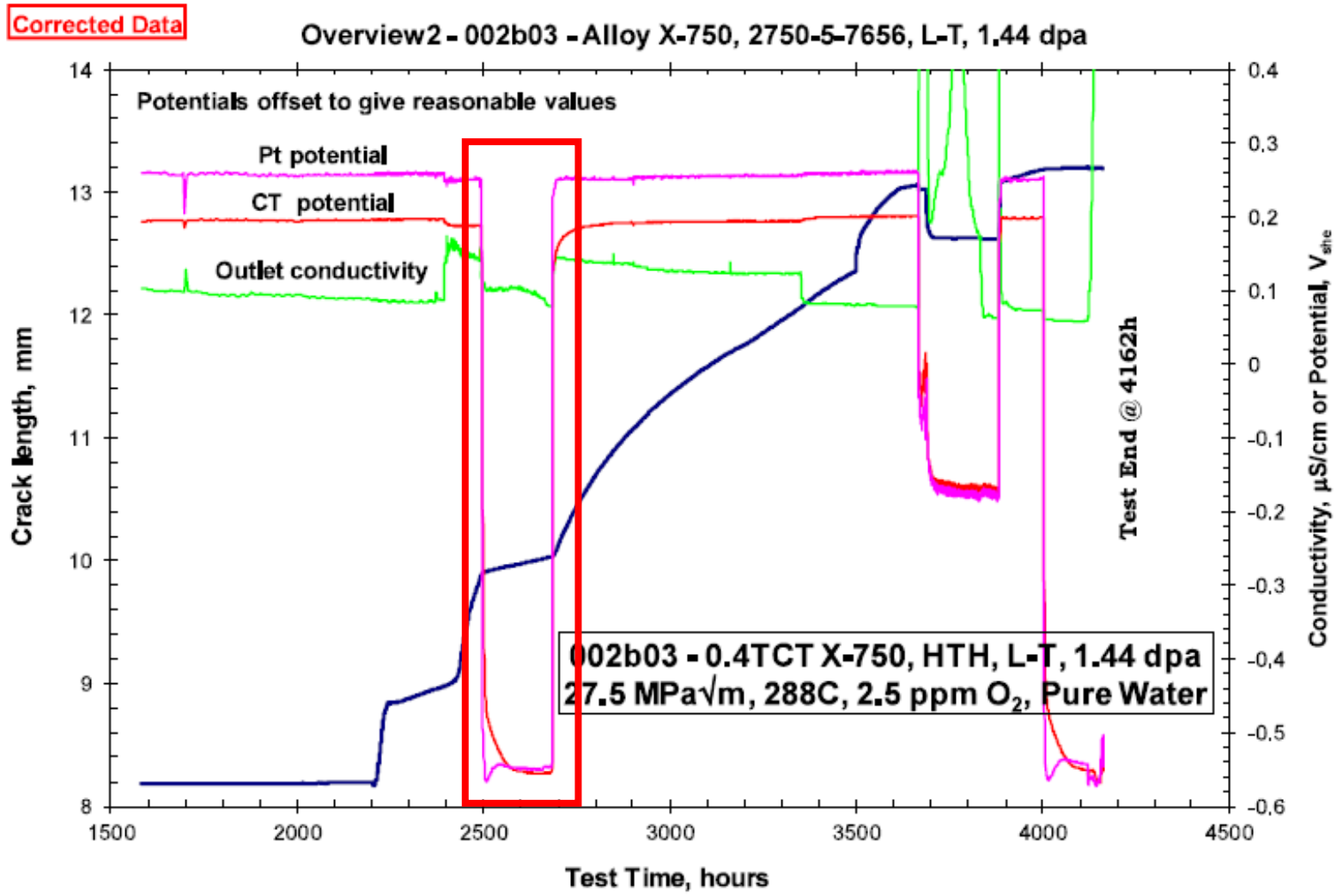
# X-750 ~0.3 dpa CGR (02A10) - Results

| Test Hours | K (MPa√m) | Chemistry | Sulfate | Outlet Cond (µS/cm) | Time Increment (hr) | Growth Increment (mm) | Average CGR (mm/s) |
|---|---|---|---|---|---|---|---|
| 3146 | 28 | NWC | 10 | 0.131 | 41 | 0.394 | 2.60E-06 |
| 3187 | 29 | HWC | 10 | 0.062 | 212 | 0.112 | 3.60E-08 |
| 3399 | 29 | NWC | 10 | 0.15 | 20 | 0.032 | 1.00E-06 |
| 3444 | 18 | NWC | 10 | 0.13 | 79 | 0.130 | 5.00E-07 |
| 3523 | 18 | HWC | 10 | 0.095 | 361 | 0.023 | 7.40E-09 |
| 3884 | 18 | NWC | 10 | 0.13 | 137 | 0.456 | 5.50E-07 |
| 4077 | 30 | NWC | 10 | 0.13 | 43 | 0.161 | 1.10E-06 |
| 4120 | 30 | HWC | 10 | 0.08 | 335 | 0.048 | 1.90E-08 |
| 4532 | 30 | NWC | 10 | 0.15 | 188 | 0.278 | 5.80E-07 |
| 4730 | 49 | NWC | 10 | 0.145 | 62 | 0.132 | 6.50E-07 |
| 4792 | 50 | HWC | 10 | 0.085 | 172 | 0.041 | 6.50E-08 |

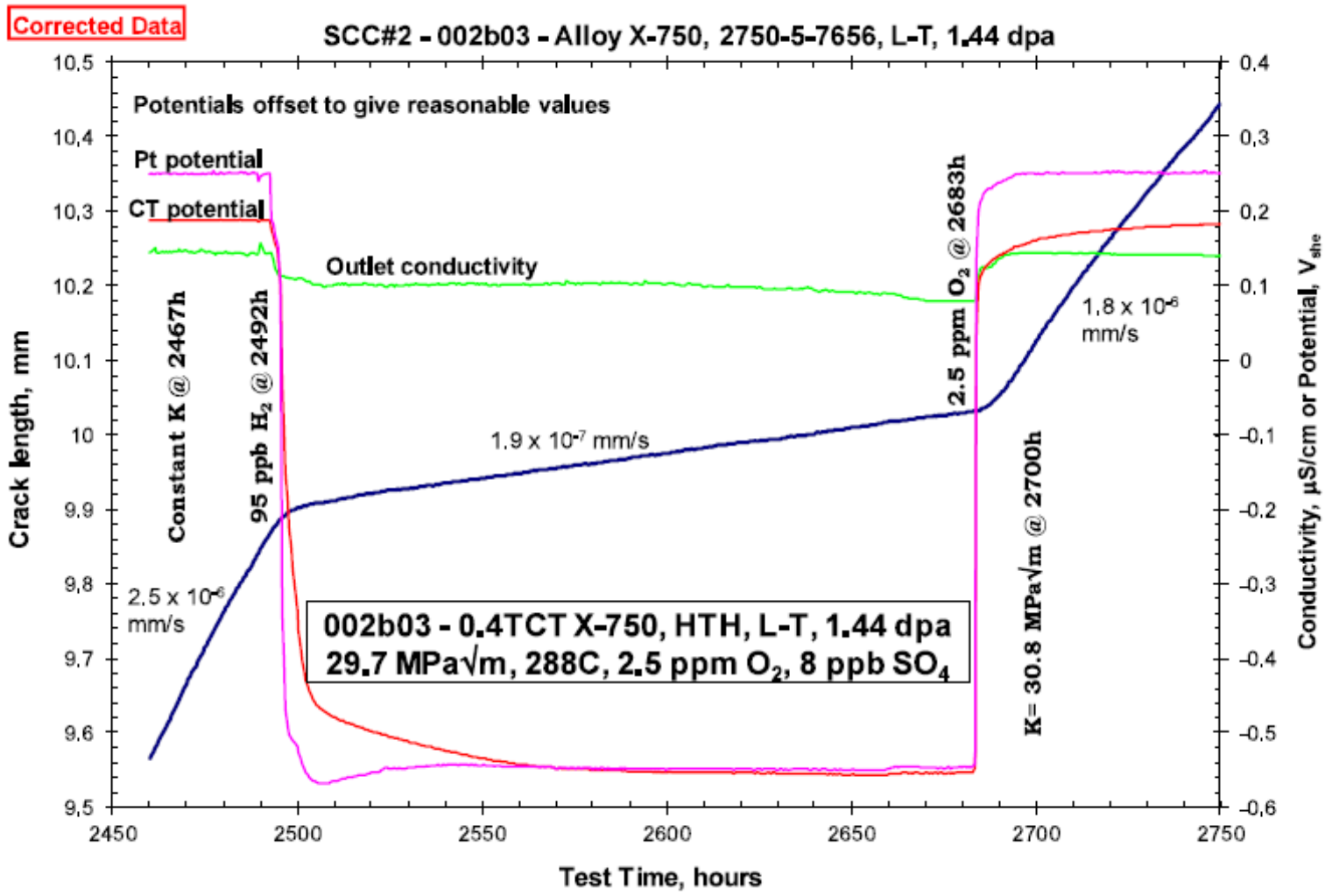



rows highlighted in blue denote measurements which were taken at a high degree of confidence

# X-750 ~1.4 dpa CGR (02B03) – Overview of CGR Test

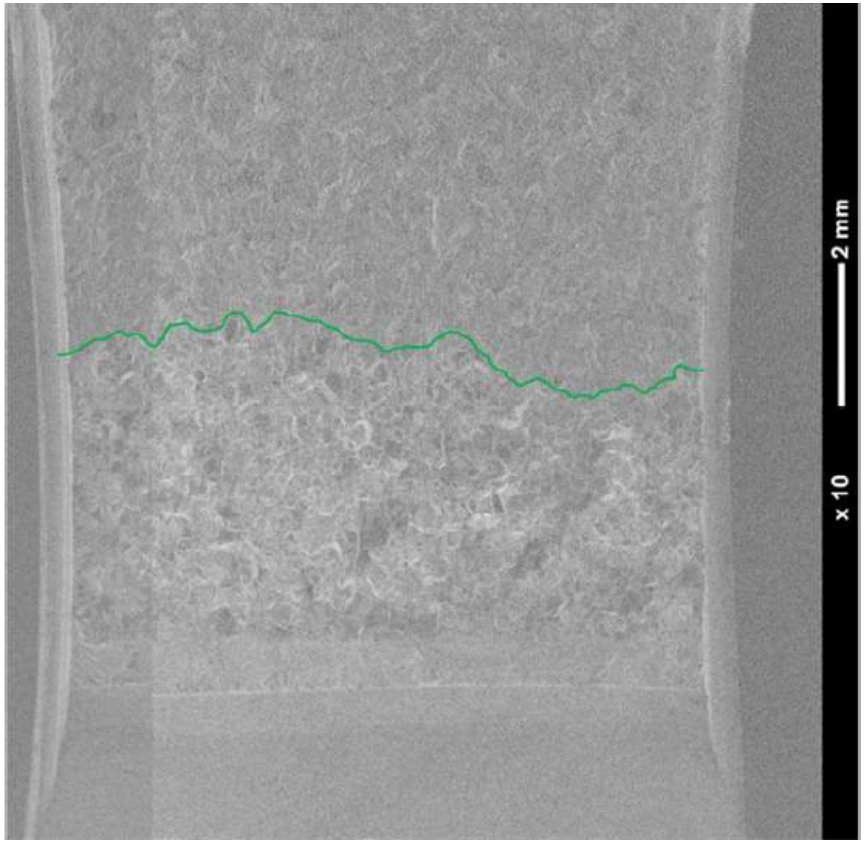


# X-750 ~1.4 dpa CGR (02B03) – Selected Evaluation Step



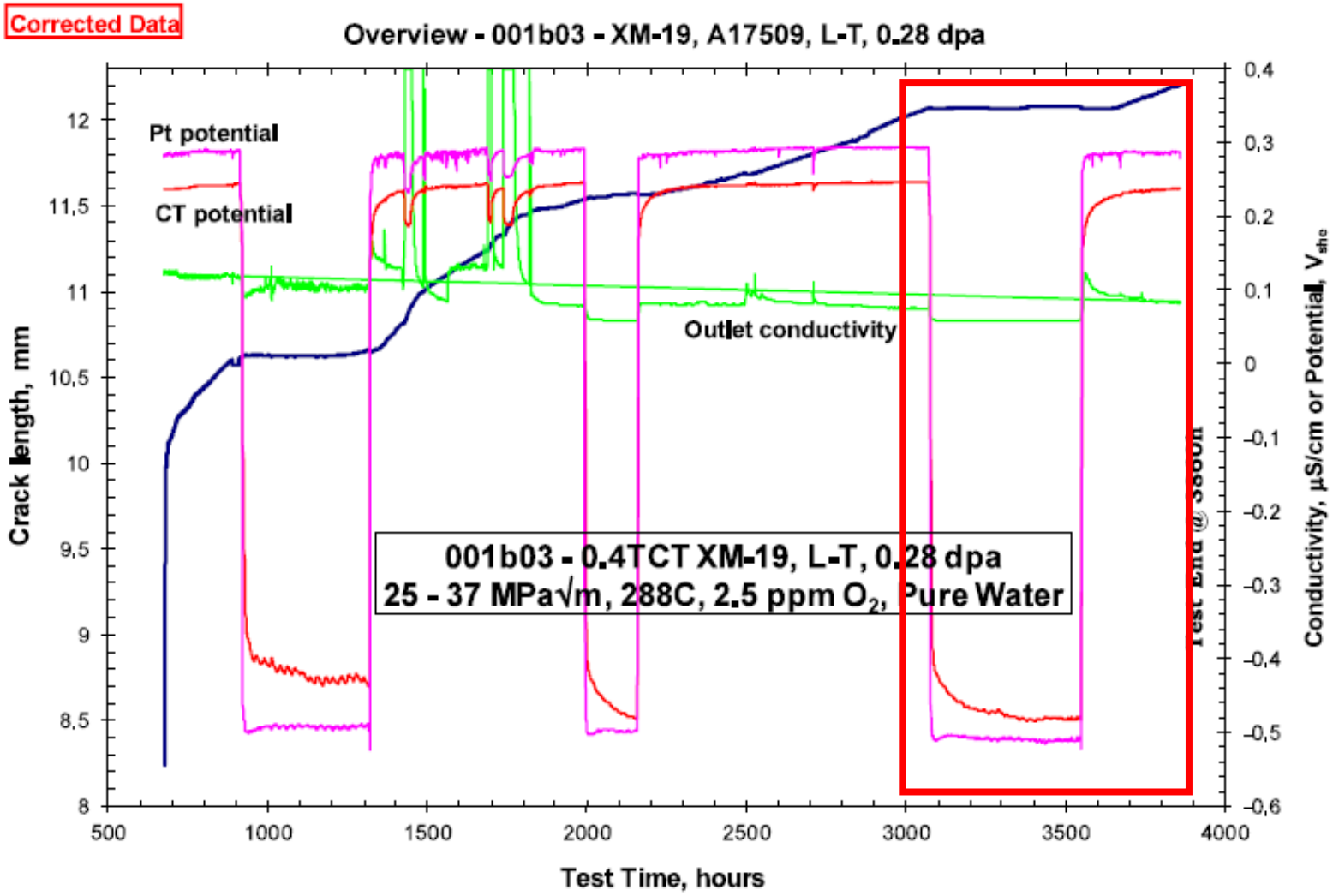
# X-750 ~1.4 dpa CGR (02B03) – Results

| Test Hours | K (MPa√m) | Chemistry | Sulfate | Outlet Cond (µS/cm) | Time Increment (hr) | Growth Increment (mm) | Average CGR (mm/s) |
|---|---|---|---|---|---|---|---|
| 2467 | 29 | NWC | 8 | 0.148 | 25 | 0.232 | 2.50E-06 |
| 2492 | 30 | HWC | 8 | 0.1 | 191 | 0.160 | 1.90E-07 |
| 2683 | 31 | NWC | 8 | 0.141 | 70 | 0.428 | 1.80E-06 |
| 3174 | 22 | NWC | 8 | 0.123 | 177 | 0.346 | 5.20E-07 |
| 3351 | 23 | NWC | 0 | 0.082 | 146 | 0.291 | 4.50E-07 |
| 3887 | 24 | NWC | 0 | 0.073 | 115 | 0.104 | 2.60E-07 |
| 4002 | 26 | HWC | 0 | 0.06 | 151 | 0.013 | 3.60E-08 |

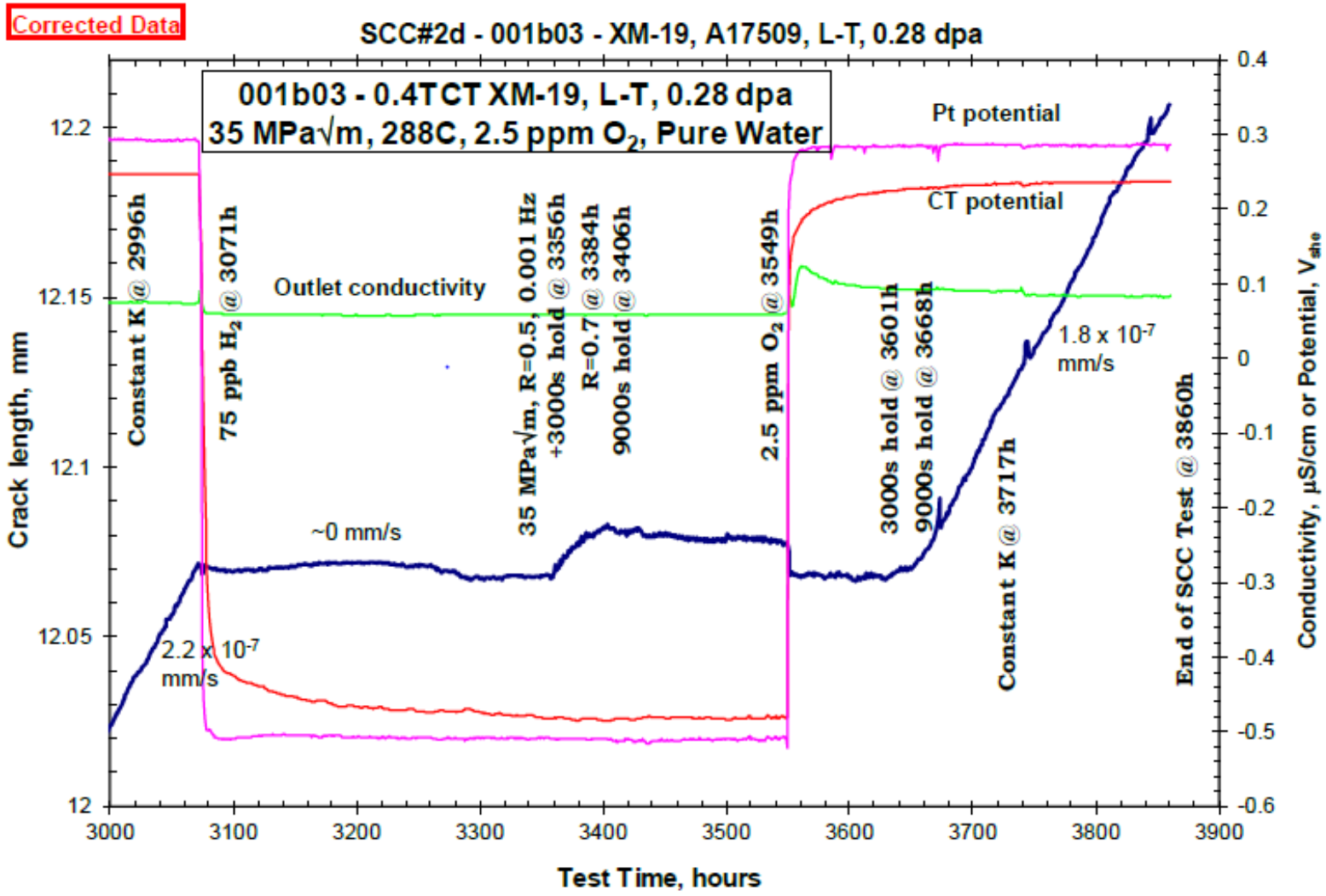



rows highlighted in blue denote measurements which were taken at a high degree of confidence

# XM-19 ~0.3 dpa CGR (01B03) – Overview of CGR Test

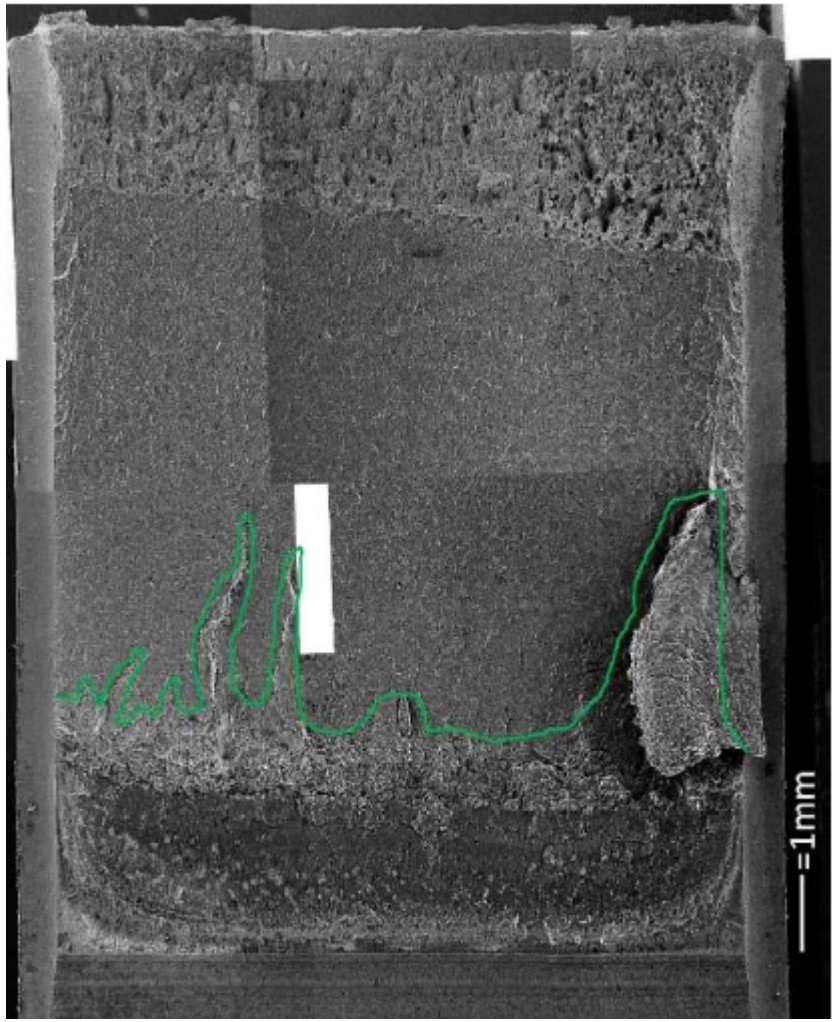


# XM-19 ~0.3 dpa CGR (01B03) – Selected Evaluation Step



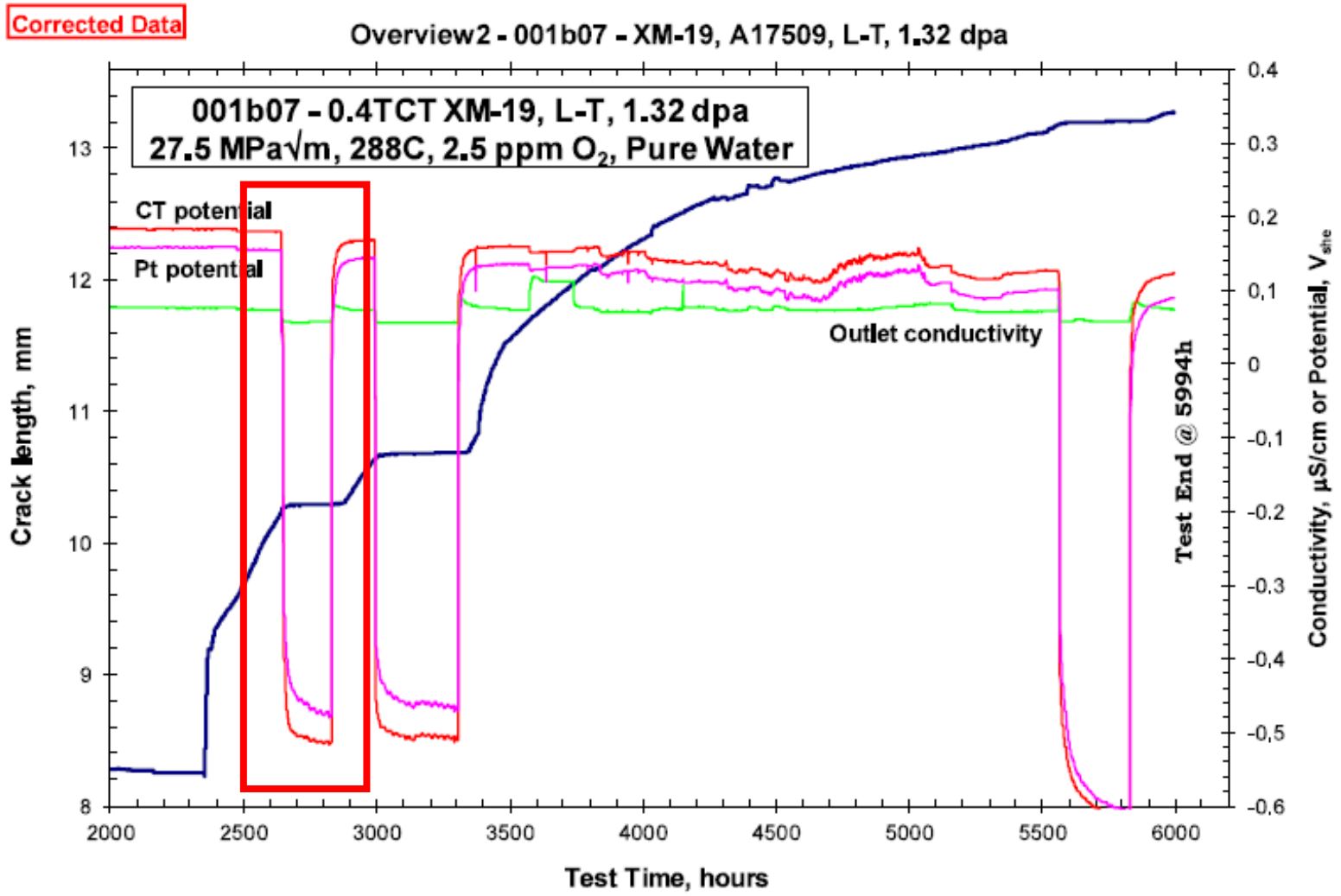
# XM-19 ~0.3 dpa CGR (01B03) - Results

| Test Hours | K (MPa√m) | Chemistry | Sulfate | Outlet Cond (µS/cm) | Time Increment (hr) | Growth Increment (mm) | Average CGR (mm/s) |
|---|---|---|---|---|---|---|---|
| 1537 | 33 | NWC | 0 | 0.087 | 24 | 0.028 | 3.20E-07 |
| 1561 | 33 | NWC | 10 | 0.129 | 261 | 0.362 | 3.20E-07 |
| 1844 | 33 | NWC | 0 | 0.08 | 148 | 0.059 | 1.60E-07 |
| 1992 | 33 | HWC | 0 | 0.059 | 69 | 0.015 | 2.00E-08 |
| 2540 | 35 | NWC | 0 | 0.081 | 314 | 0.193 | 1.80E-07 |
| 2996 | 35 | NWC | 0 | 0.075 | 79 | 0.049 | 2.20E-07 |
| 3076 | 35 | HWC | 0 | 0.059 | 280 | 0.000 | 1.00E-09 |
| 3717 | 35 | NWC | 0 | 0.083 | 143 | 0.092 | 1.80E-07 |

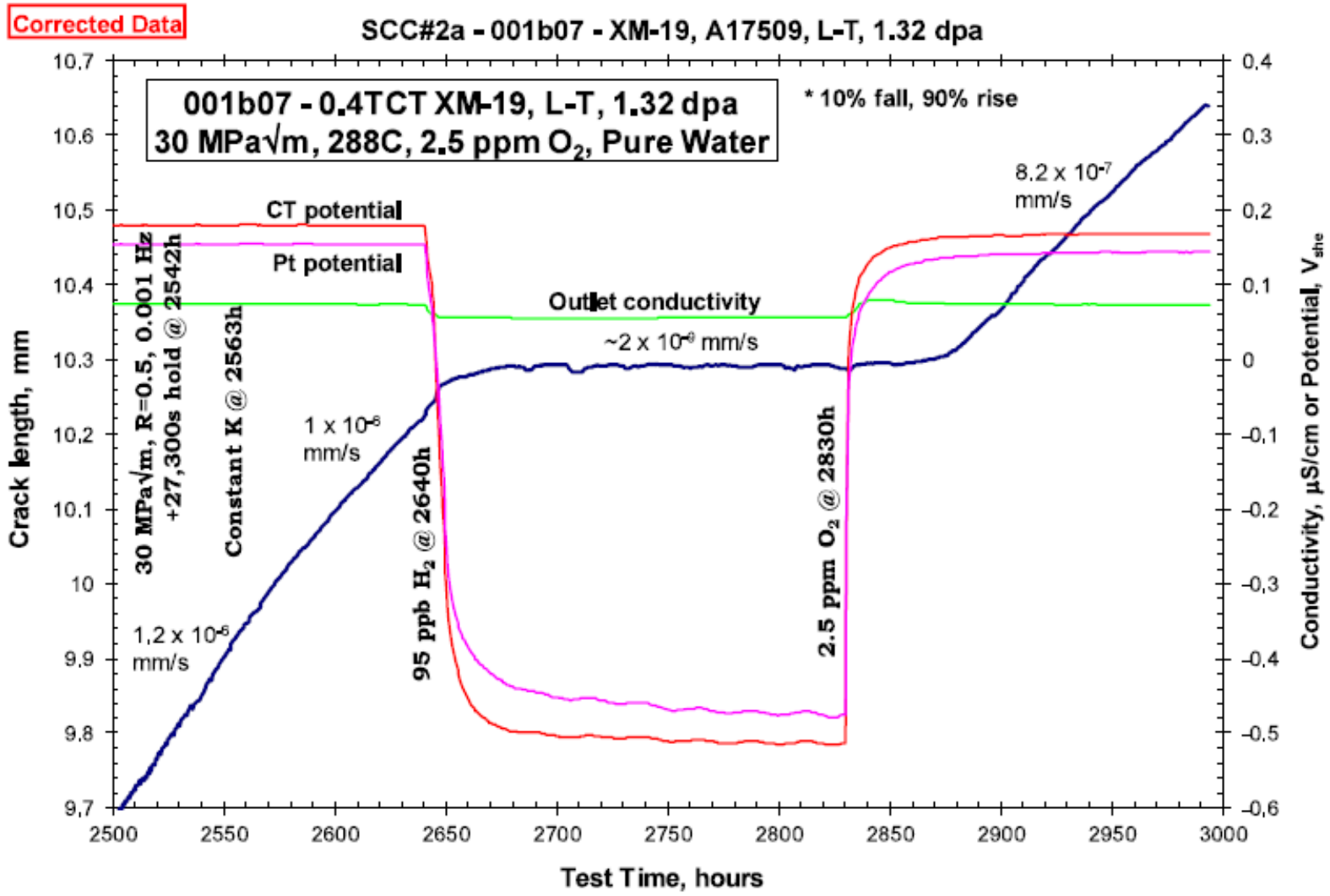



rows highlighted in blue denote measurements which were taken at a high degree of confidence

# XM-19 ~1.5 dpa CGR (01B07) – Overview of CGR Test

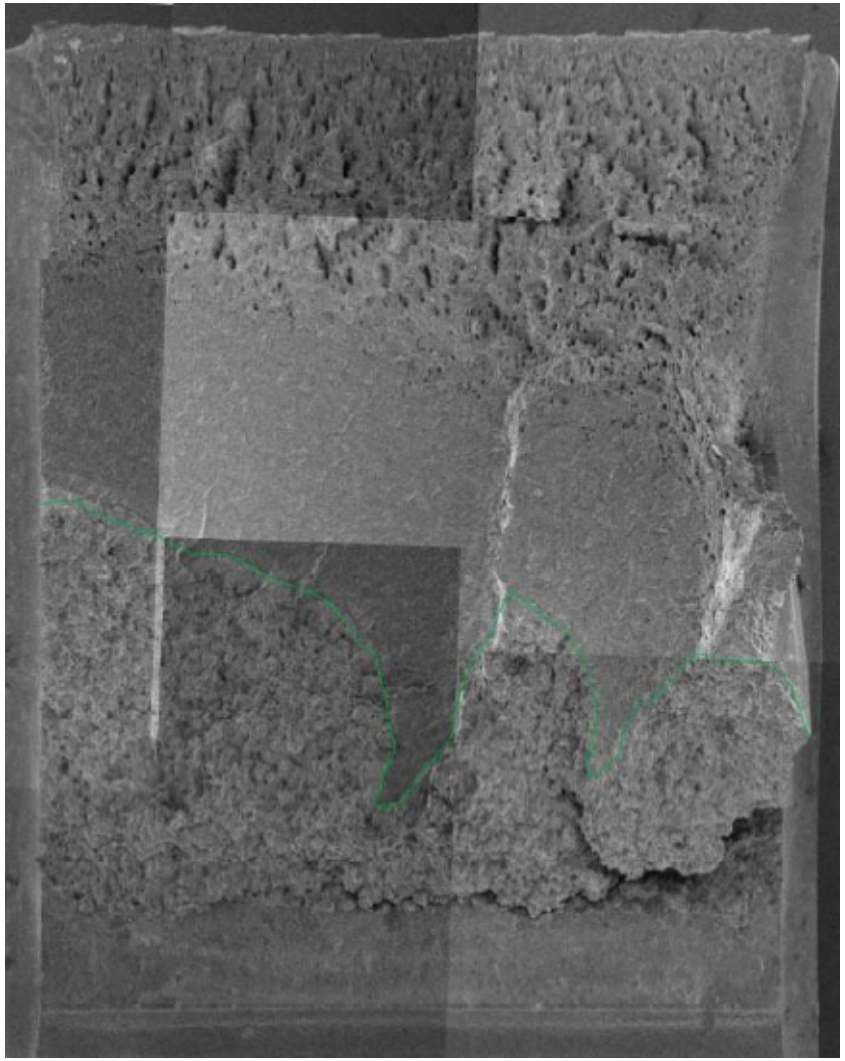


# XM-19 ~1.4 dpa CGR (01B07) – Selected Evaluation Step



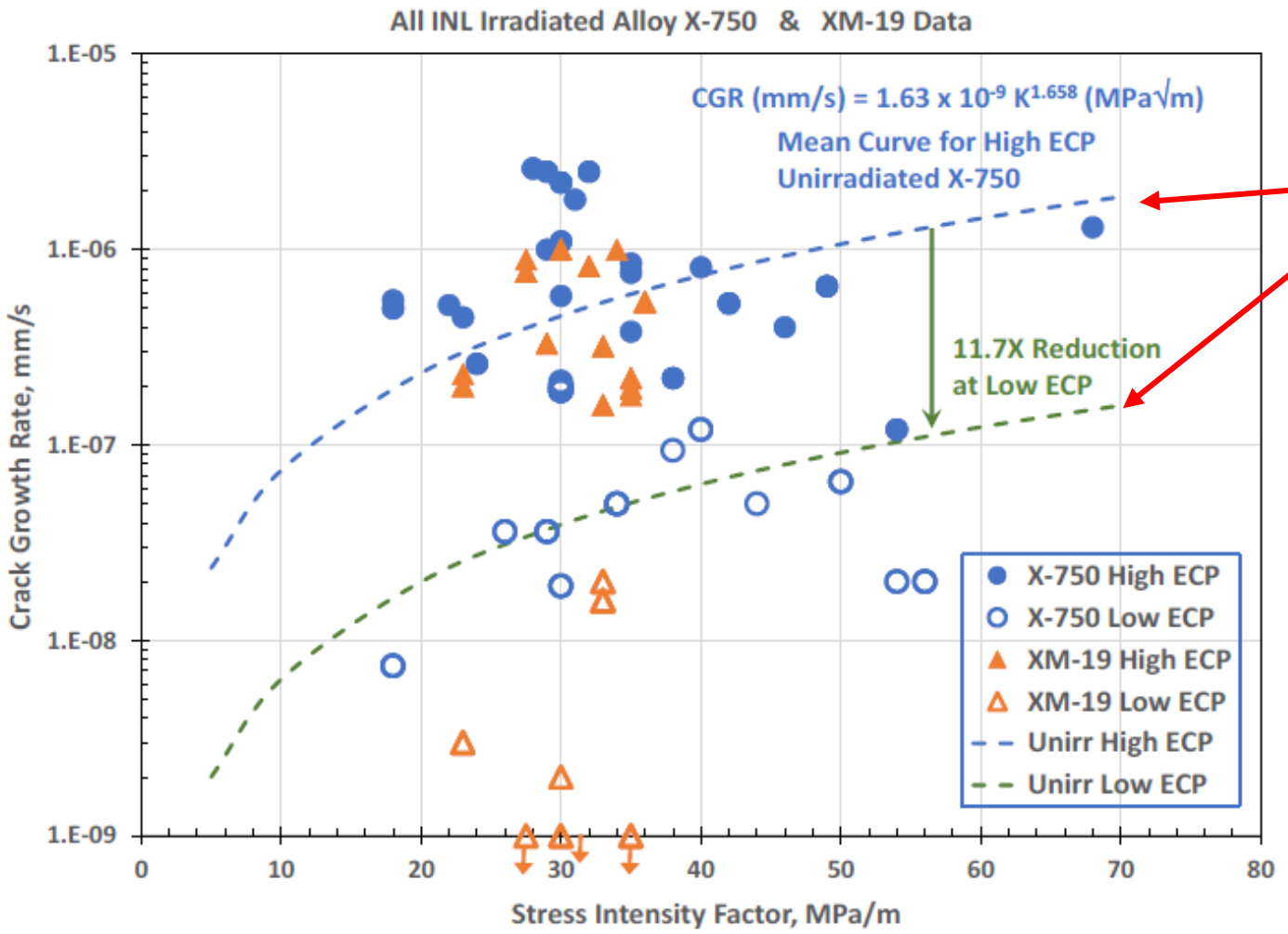
# XM-19 ~1.4 dpa CGR (01B07) – Results

| Test Hours | K (MPaVm) | Chemistry | Sulfate | Outlet Cond (µS/cm) | Time Increment (hr) | Growth Increment (mm) | Average CGR (mm/s) |
|---|---|---|---|---|---|---|---|
| 2563 | 30 | NWC | 0 | 0.074 | 77 | 0.263 | 1.00E-06 |
| 2640 | 30 | HWC | 0 | 0.058 | 190 | 0.063 | 2.00E-09 |
| 2830 | 32 | NWC | 0 | 0.073 | 164 | 0.355 | 8.20E-07 |
| 2994 | 33 | HWC | 0 | 0.056 | 312 | 0.047 | 1.60E-08 |
| 3306 | 34 | NWC | 0 | 0.078 | 76 | 0.150 | 1.00E-06 |
| 3480 | 36 | NWC | 0 | 0.074 | 92 | 0.176 | 5.40E-07 |
| 3572 | 36 | NWC | 8 | 0.113 | 243 | 0.389 | 5.40E-07 |
| 3815 | 36 | NWC | 0 | 0.07 | 164 | 0.204 | 5.40E-07 |
| 5213 | 23 | NWC | 0 | 0.072 | 350 | 0.176 | 2.00E-07 |
| 5563 | 23 | HWC | 0 | 0.057 | 265 | 0.024 | 3.00E-09 |



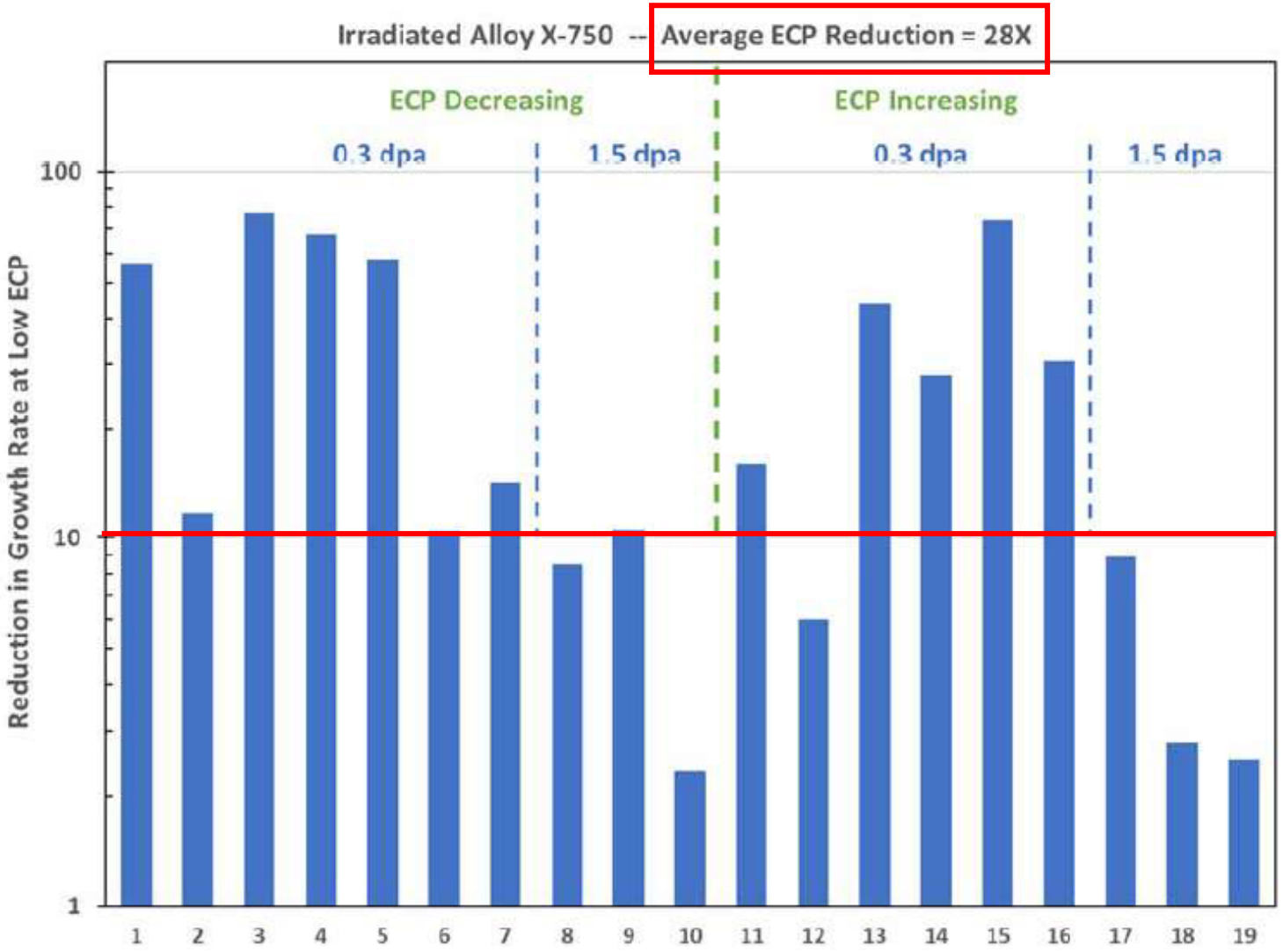
rows highlighted in blue denote measurements which were taken at a high degree of confidence

# Compiled CGR Data for Neutron Irradiated X-750 and XM-19



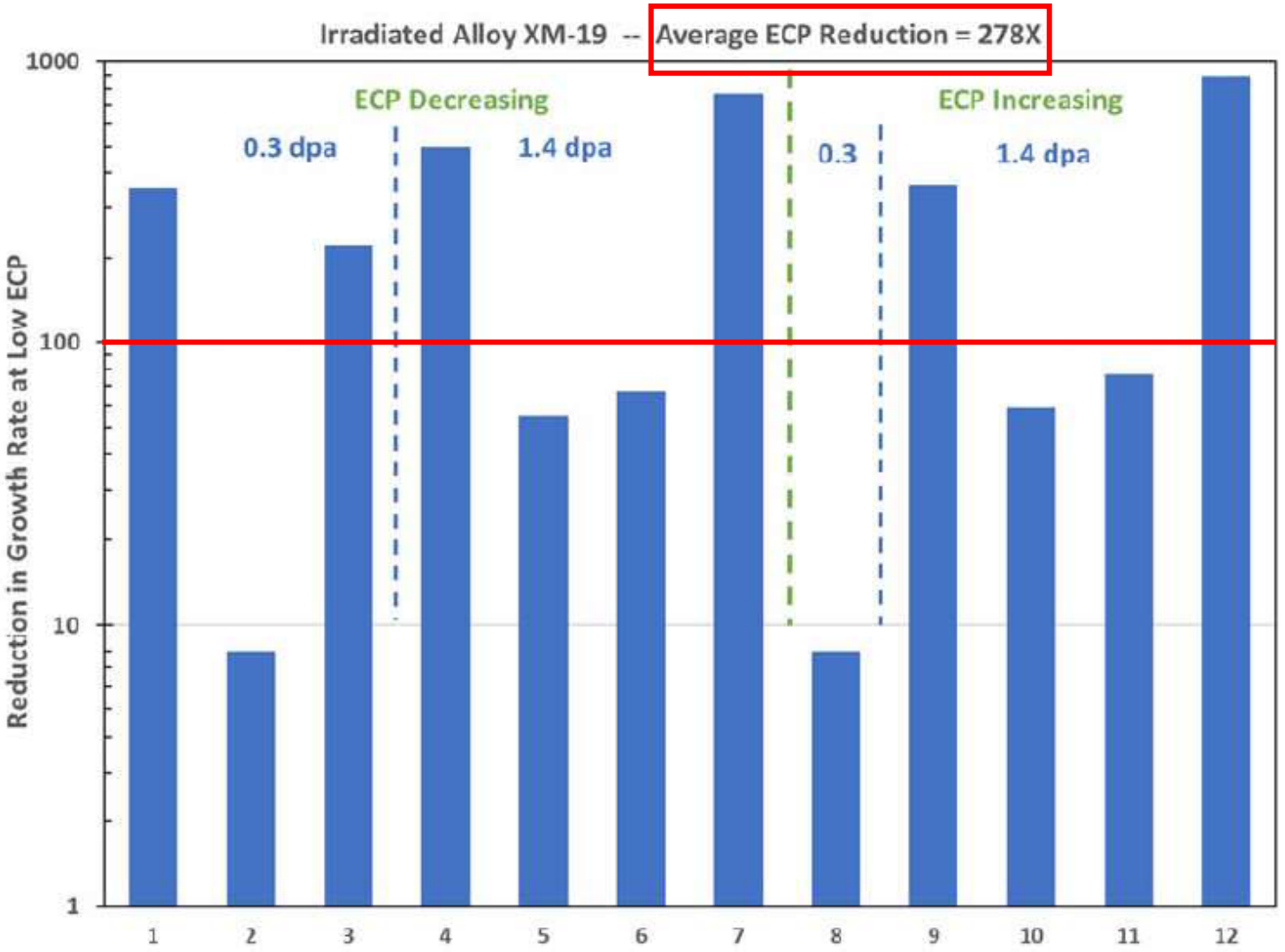

Dashed lines represent curves generated from non-irradiated data

# Effect of Low/High ECP on X-750 CGR



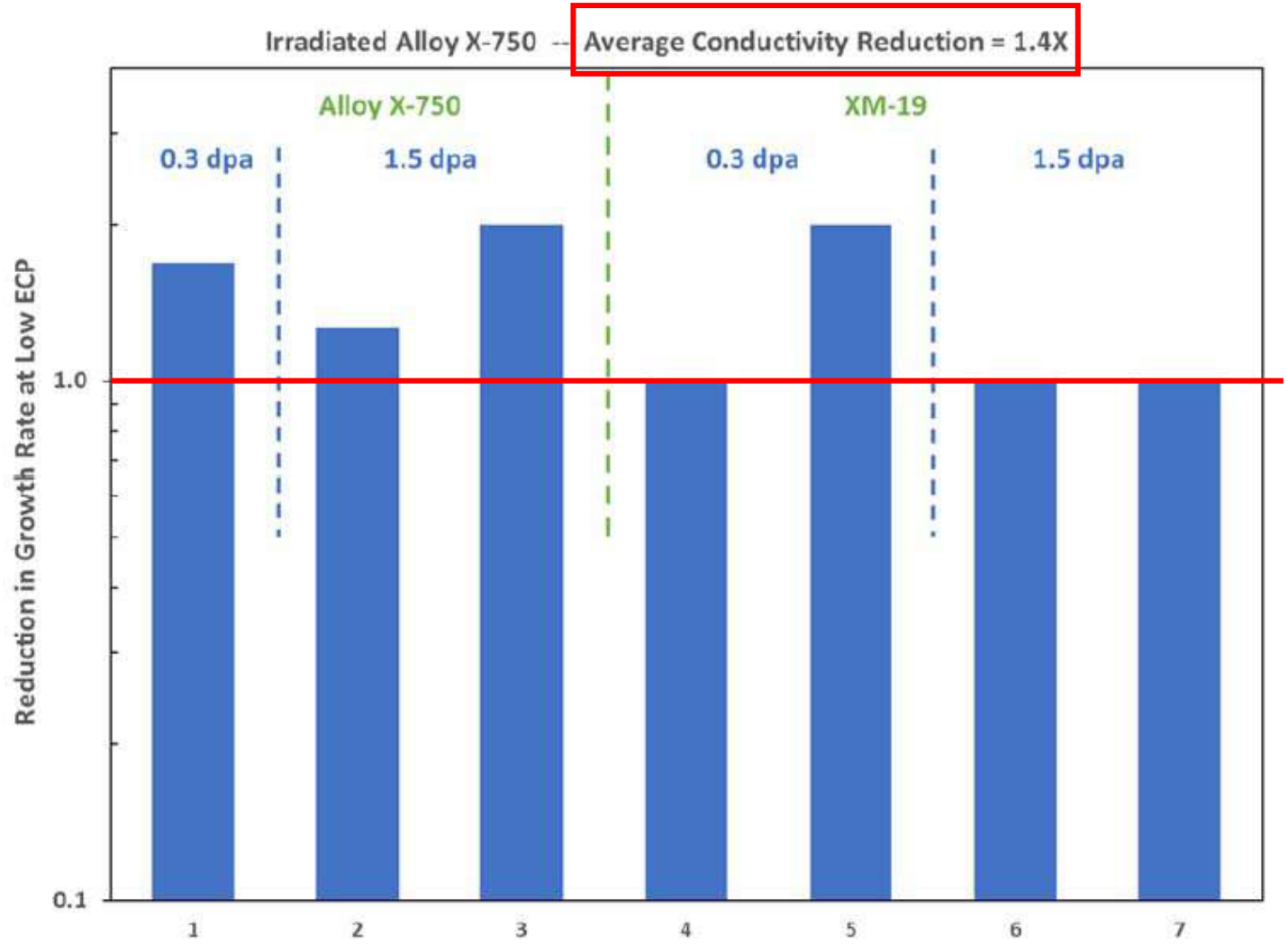

Data represents results from both ~0.3 dpa and ~1.4 dpa samples

# Effect of Low/High ECP on XM-19 CGR



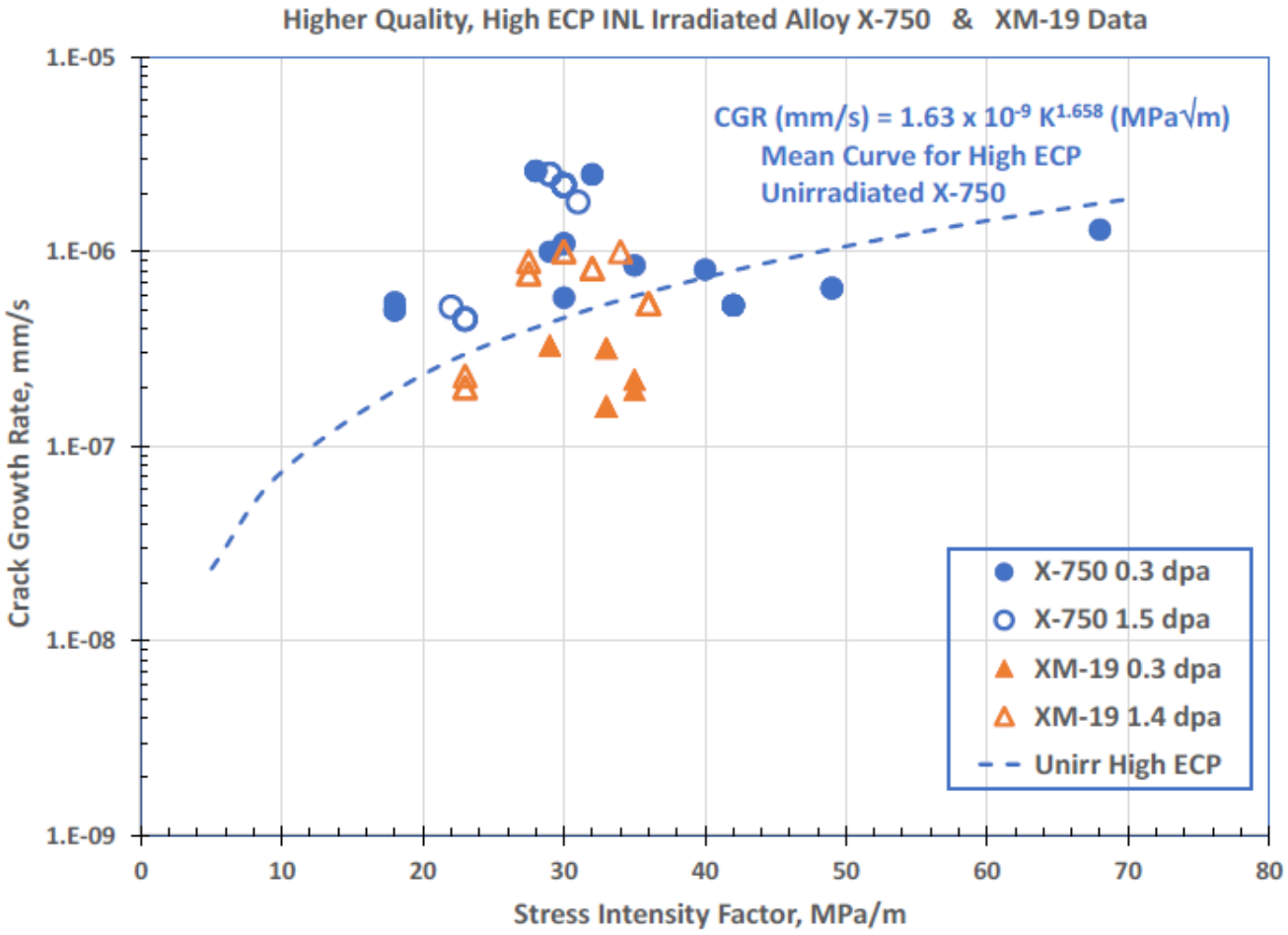

Data represents results from both ~0.3 dpa and ~1.4 dpa samples

# Effects of Sulfate Additions on X-750 and XM-19 CGR

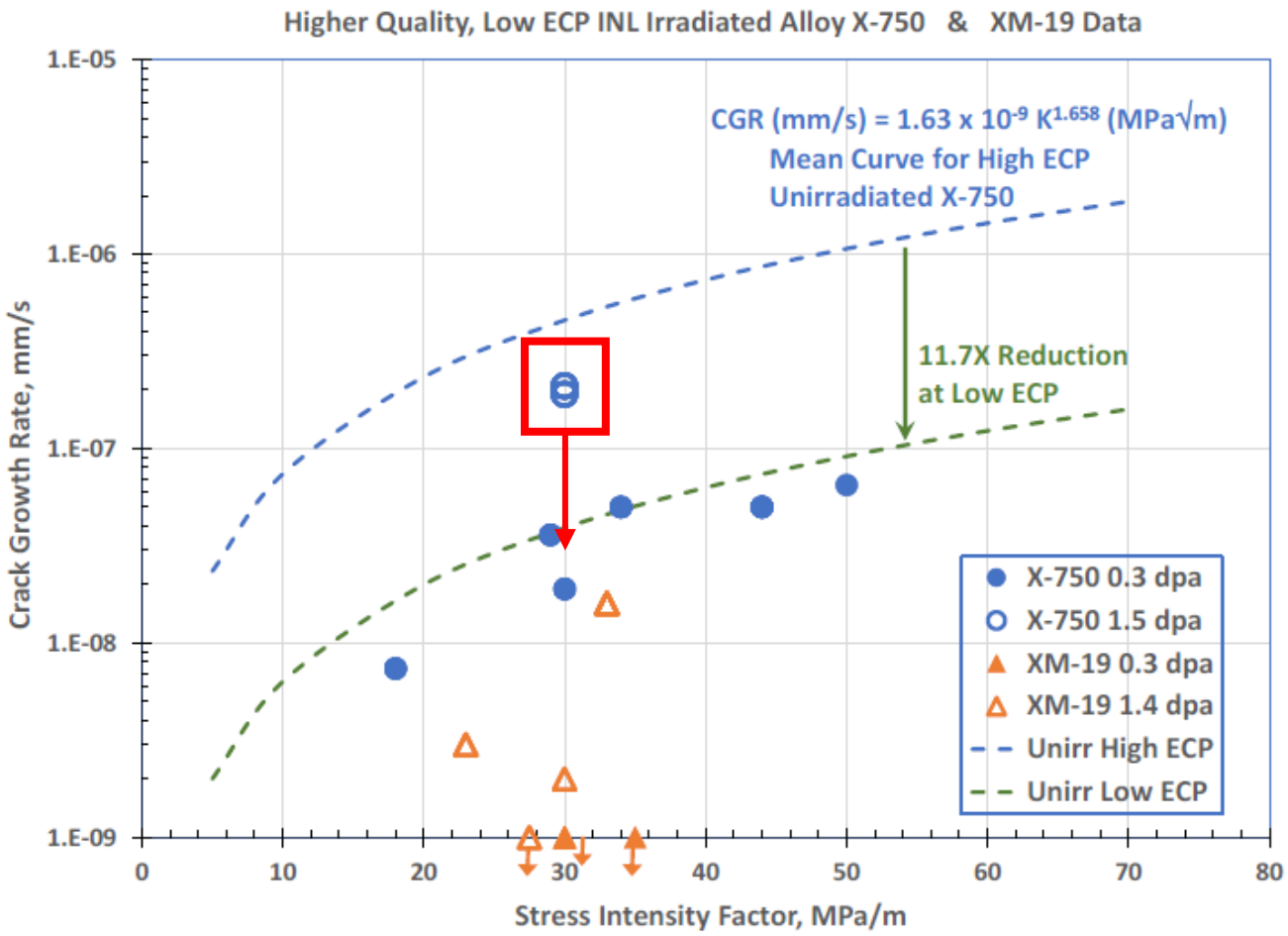



Data represents results from both ~0.3 dpa and ~1.4 dpa samples

# Effect of Dose on CGR in Neutron Irradiated X-750 and XM-19 (High ECP)



# Effect of Dose on CGR in Neutron Irradiated X-750 and XM-19 (Low ECP)



# Conclusion

- First of its kind experiment for the civilian sector performed in the ATR CFT
- Significant changes in CGR were observed for neutron irradiated X-750 and XM-19 after switching between NWC and HWC environments (28x for X-750, 278x for XM-19)
- Slight effect (~1.4x) on CGR observed after changing between 10 ppb sulfate and pure water
- Overall CGR of irradiated samples was similar to non-irradiated CGR data, supplying evidence that the CGR of these alloys show limited sensitivity to dose
- Effect of dose on the CGR in neutron irradiated X-750 and XM-19 was only distinct in the low ECP environment, however the very low growth rates observed in HWC for these alloys reduces the confidence of this conclusion