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Stress Corrosion Cracking Initiation of Ni-Based Alloys in High Temperature Water

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Stress Corrosion Crack Initiation Program

- Objective: Provide Stress corrosion cracking (SCC) initiation data for Alloy 600 that is not compromised by:
  - stress relaxation
  - an unknown stress state
  - position relative to Ni/NiO phase transition
  - uncertainty in time to initiation
- Test matrix designed to study the effects of temperature and coolant hydrogen concentration.
- Method: Instrumented smooth tensile specimens actively loaded.

The goal of the work is to provide stress corrosion cracking initiation data for Alloy 600 that is not compromised by 1) specimens that suffer from stress relaxation, 2) specimens which have an unknown stress state, 3) specimens which are tested at unknown positions electrochemically relative to the Ni/NiO phase transition, and 4) testing which relies on the period of time between specimen inspection intervals to estimate SCC initiation times.

The current study was aimed at studying the effects of temperature and coolant hydrogen concentration on SCC initiation in high purity, high temperature water.

Instrumented, smooth tensile specimens that are actively loaded are used in order to test a specimen with a known and controlled stress state.

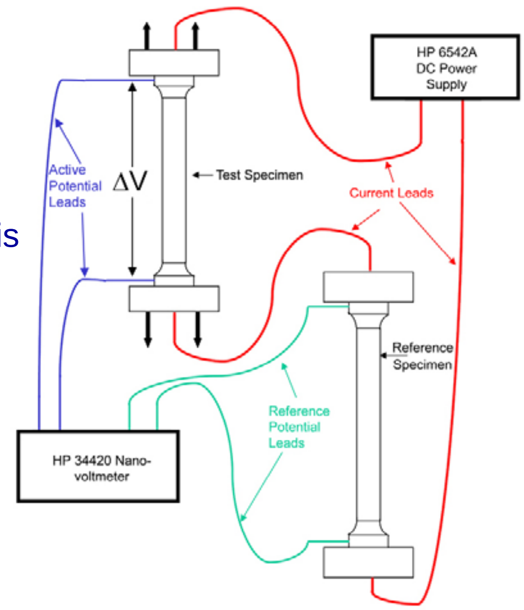
DC Electrical Potential Drop (EPD) to Measure Initiation

$R = \Delta V/I = \rho L/A$

In Plastic Regime Volume is Constant ($A=V_o/L$)

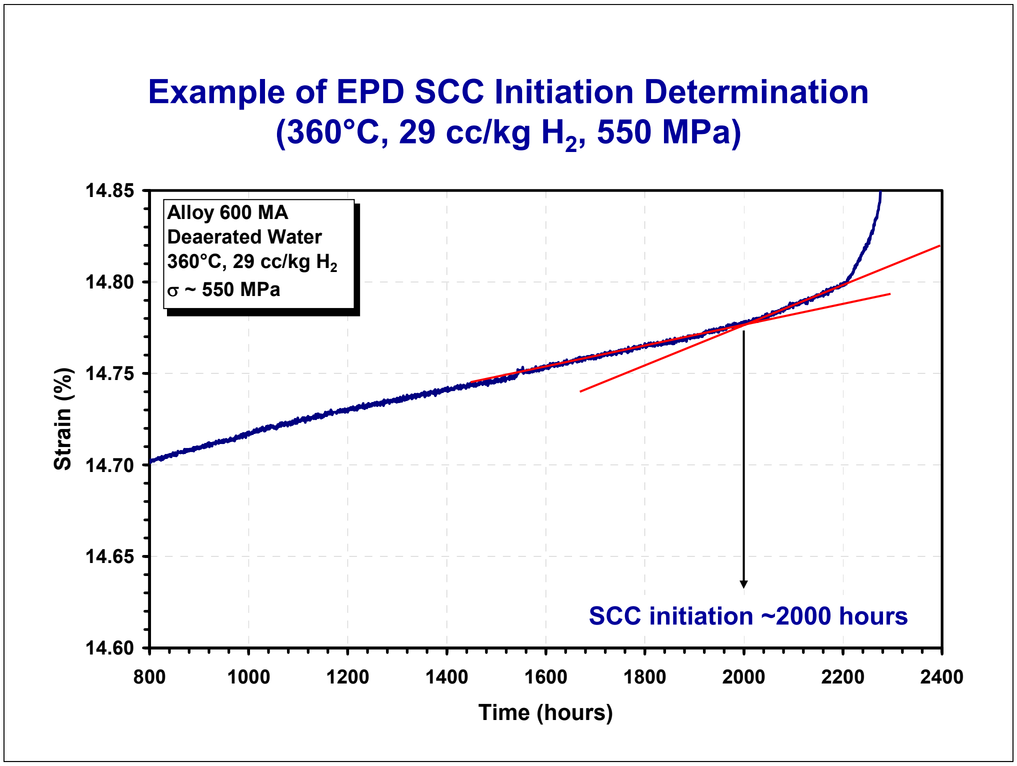
$R = (\rho/V_o)(L_o+\Delta L)^2 = (\rho L_o^2/V_o)(1+\Delta L/L_o)^2$

Strain, $\varepsilon = (R/R_o)^{1/2} - 1$

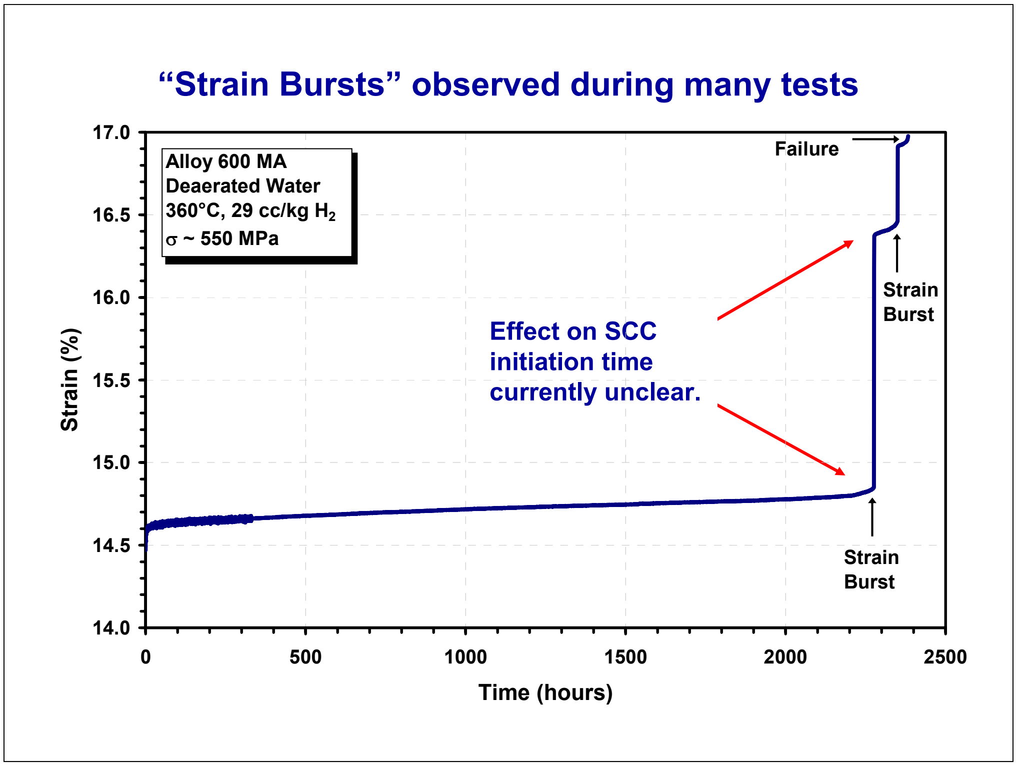


∴ Strain is related to change in resistance due to SCC initiation

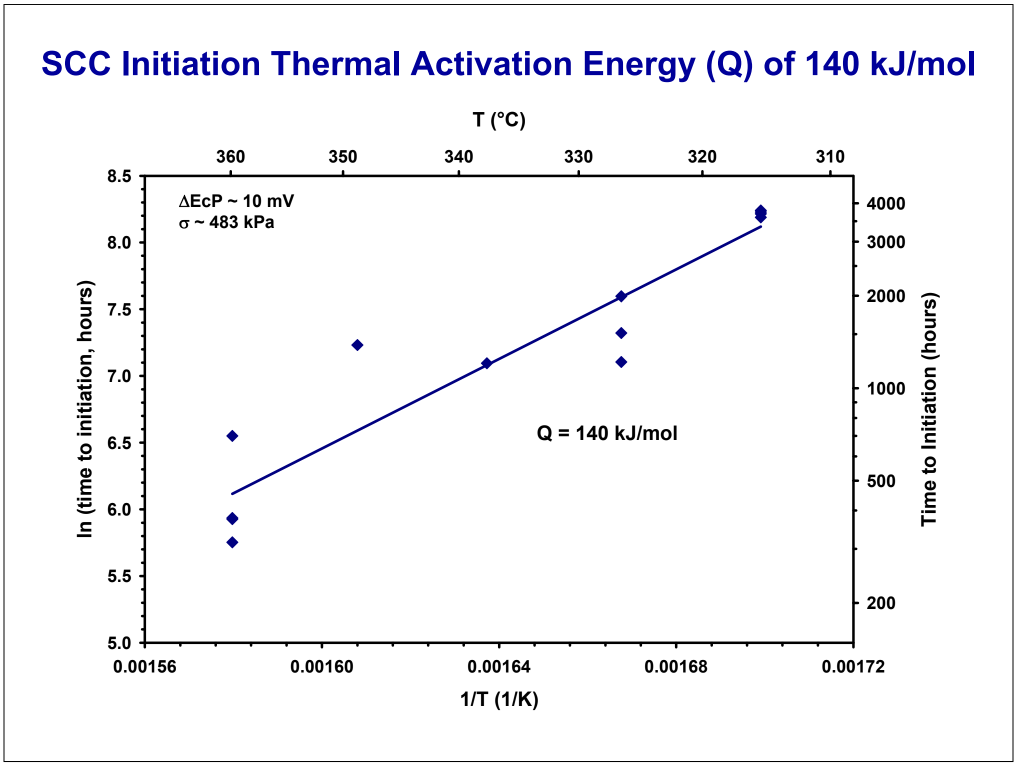
DC electric potential drop is used to measure the stress corrosion cracking (SCC) initiation time. A constant DC current is passed through the specimen and the voltage difference between probes on each end of the gage length is measured. The current distribution is uniform, thus the voltage difference between the two probes is proportional to the distance between the probes and inversely proportional to the cross-sectional area of the gage region. Formation of SCC reduces the gage cross-sectional area and causes an increase in the voltage difference between the two probes. This increase in voltage signifies SCC initiation. The voltage measured on the test specimen is normalized for temperature and material resistivity changes with a voltage measured from a “reference” specimen of the same material. The “reference” specimen is pre-strained at the test temperature to the same load as the test specimen prior to testing, but it is not under any load during the test. SCC has not occurred in any of these non-loaded pre-strained specimens.



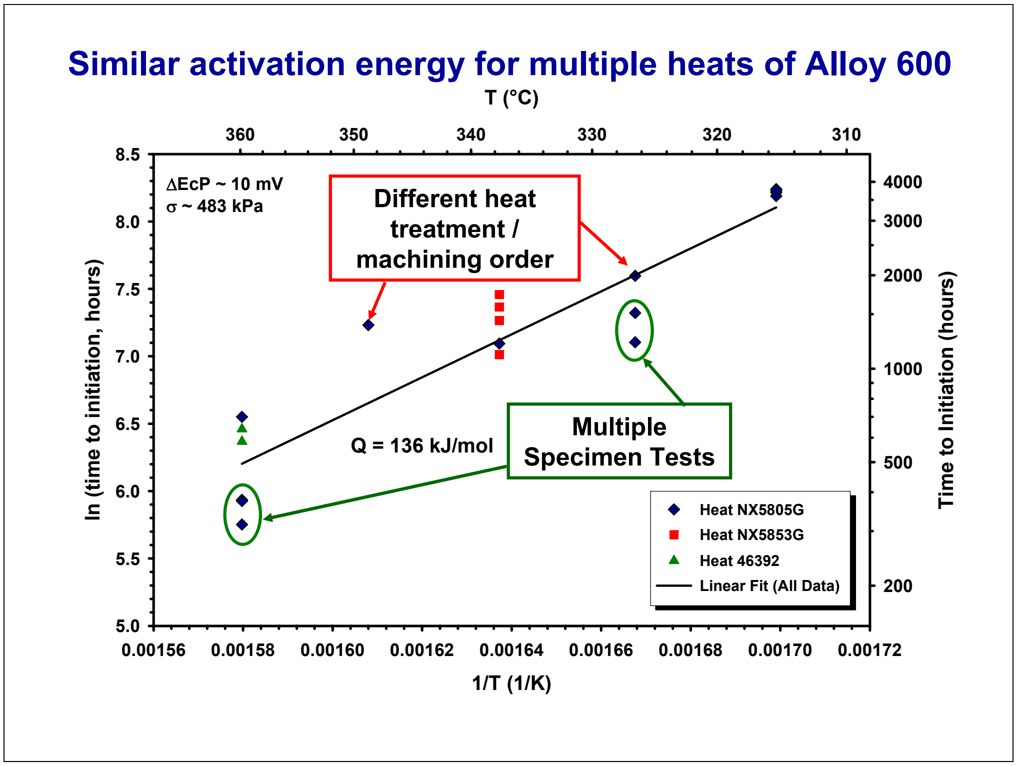
Here is an example of the use of EPD to detect SCC initiation in an Alloy 600 sample exposed to high purity water with 29 cc/kg hydrogen at 360°C. The sample was loaded to a stress of 550 MPa. As illustrated, lines were drawn over ranges of EPD data both before and after the slope of the EPD versus time data was noted to change. The intersection of these lines was defined as the time at which SCC initiated. In this case, initiation occurred at ~2000 hours. Slope changes were not observed in the EPD data for the tests where SCC did not occur.



Strain bursts were observed in the active load tests. Strain bursts are instantaneous increases in the strain of the specimen and were easily detected with EPD and the actuator position. Strain bursts have been reported by other researchers for Ni-Cr-Fe alloys (e.g., Alloy 690, Alloy 618, etc.) during creep testing at temperatures between 454 and 566°C, as well as in austenitic stainless steels. Commercial literature tentatively attributed strain bursts to microstructural reordering, and the effect of strain bursts, if any, on SCC initiation is not yet understood. One active-load test was conducted to determine if a strain burst would initiate SCC; SCC was not observed in the specimen following the first observed strain burst.



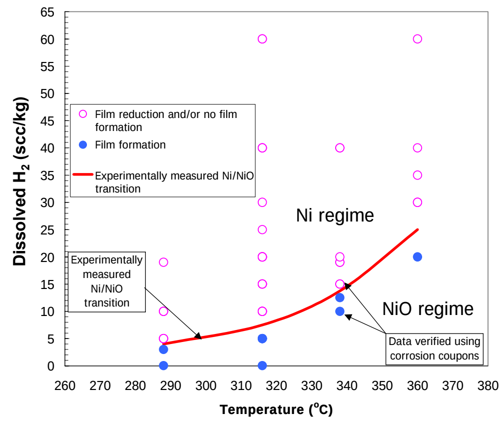
Tests were conducted at a constant electrochemical potential distance from the Ni/NiO phase transition in order to determine the activation energy for SCC initiation. The thermal activation energy (Q) for solution annealed Alloy 600 SCC initiation was determined based on testing at 360, 338, and 316°C. Q was determined to be 140 kJ/mol for the single Alloy 600 heat NX5805G.



Tests were conducted at a constant electrochemical potential distance from the Ni/NiO phase transition in order to determine the activation energy for SCC initiation. Included in the slide are data from heat NX5805G as well as data from two other heats (46392 and NX5853). Generally, it is a poor practice to combine multiple heats of material in a single Q evaluation since heat-to-heat variability can overwhelm the temperature dependency. Because of this, the single heat Q of 140 kJ/mol is viewed to be the best estimate for the Q of solution annealed Alloy 600 SCC initiation. However, the similarities between the single heat and combined heat Q values suggests that the solution anneal heat treatment may significantly reduce heat-to-heat variability. It is possible that SCC heat-to-heat variability is in a large part due to differences in grain boundary carbide coverage. Also, the solution anneal heat treatment essentially solubilizes grain boundary carbides, which increases the SCC susceptibility and may reduce the heat-to-heat variability.

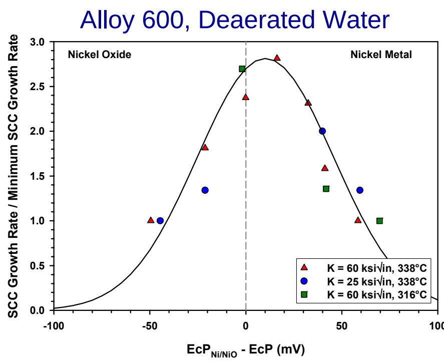
Thermal Activation Energy Studies

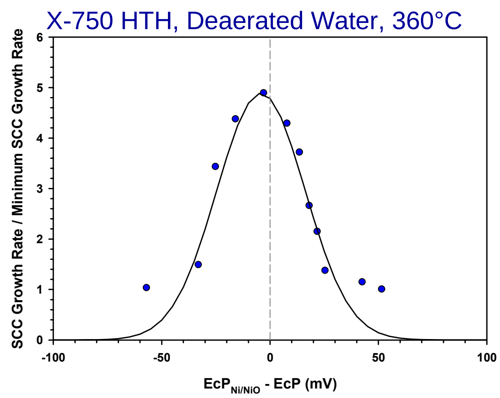
- Thermal activation energy of 140 kJ/mol less than previously reported values (e.g., 210-250 kJ/mol).
- Prior results likely biased high due to testing at constant coolant hydrogen level.
- Current testing was conducted at constant distance (ΔEcP) from the Ni/NiO phase transition at all temperatures.



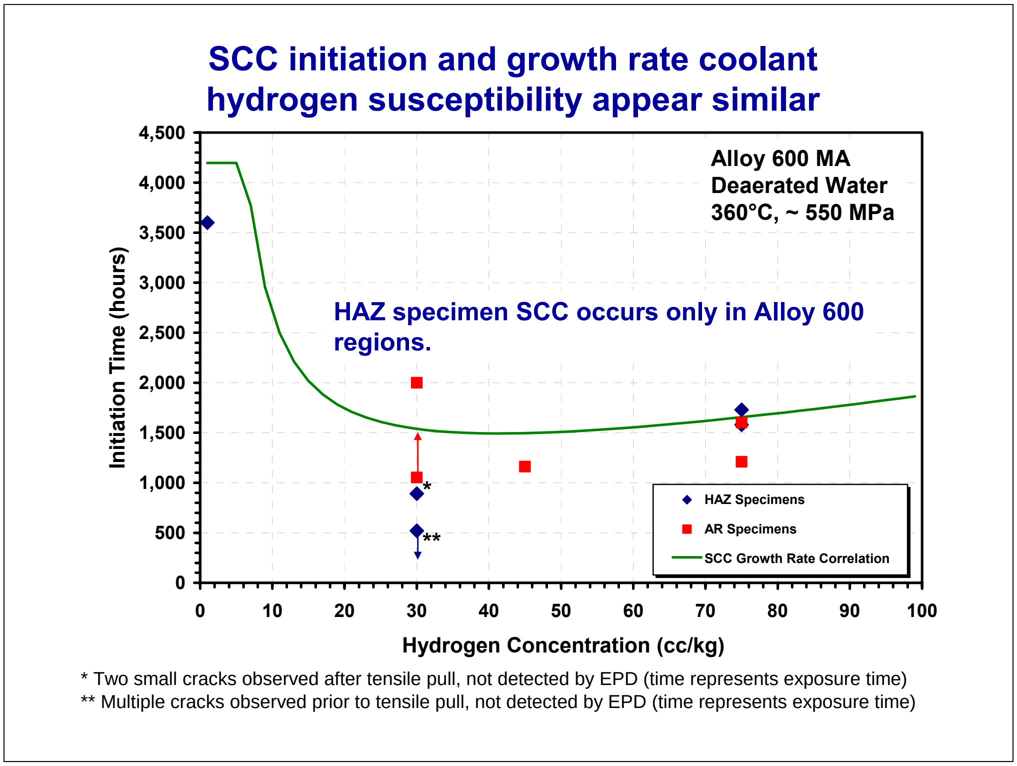
The estimated thermal activation energy of 140 kJ/mol is considerably less than values reported by other investigators. The likely explanation for this difference is the effect of coolant hydrogen. The correct methodology to conduct thermal activation energy tests for nickel alloys is to perform tests at a constant distance (in terms of ΔEcP) from the Ni/NiO phase transition at all temperatures. The tests in this study were all conducted at ΔEcP ~ 10 mV. Since the hydrogen concentration of the Ni/NiO phase transition is a function of temperature, this thermal activation energy test methodology requires that different hydrogen concentrations be tested at each temperature. However, prior to this recent coolant hydrogen understanding, thermal activation energy testing was performed at a constant coolant hydrogen level. Thermal activation energies are likely to be biased high if testing is performed at a constant coolant hydrogen level. *Thus similar to SCC crack growth rate, the true Q of SCC initiation is likely to be in the 125 to 170 kJ/mol range and higher values reported earlier (e.g., 210-250 kJ/mol) are likely biased high by coolant hydrogen effects.*

SCC Growth Rate affected by Proximity to Ni/NiO Phase Transition





Previous testing has shown that SCC growth rates in Ni alloys exposed to high temperature, high purity water are affected by the Corrosion potential distance from the nickel oxide phase transition, $\Delta EcP = (EcP_{Ni/NiO} - EcP)$. This quantity describes, fundamentally, the coolant hydrogen crack growth rate functionality.

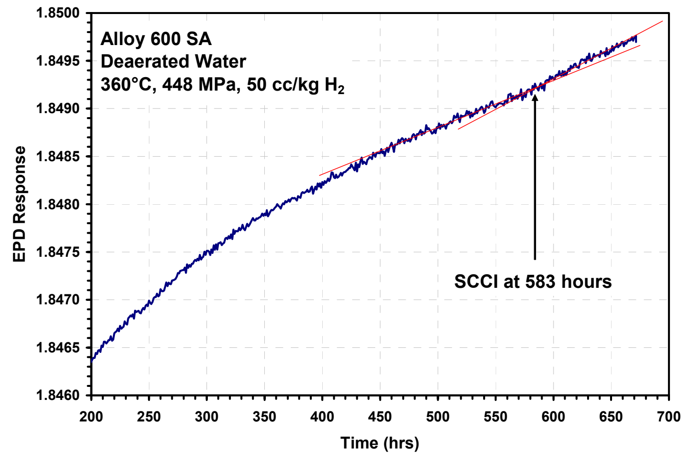


This figure summarizes the SCC initiation time as a function of coolant hydrogen for Alloy 600 heat NX5805G at 360°C. The five diamond data points on this figure represent Alloy 600 data from the HAZ specimen tests, while the four square data points correspond to pure Alloy 600 active-load test specimens. The results from the HAZ specimens are included since post test stereo-microscope specimen inspections revealed numerous Alloy 600 intergranular cracks and a lack of either EN82H weld metal or the EN82H to Alloy 600 heat affect zone cracks. Since SCC occurred only within Alloy 600 regions, the SCC initiation time data obtained with the HAZ test specimens actually represented Alloy 600 heat NX5805G behavior.

Also included is the estimated effect of coolant hydrogen on SCC initiation time. Specifically, it was assumed that the SCC initiation and crack growth rate coolant hydrogen functionalities were analogous. These limited test results suggest that the SCC initiation and SCC growth rate coolant hydrogen dependencies may be quantitatively similar. These SCC initiation coolant hydrogen results are also consistent with commercial U-bend SCC initiation tests which have shown that the SCC initiation and growth rate coolant hydrogen effects are similar.

What is Resolution of EPD?

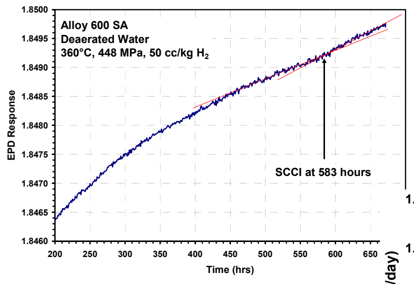
- EPD indicated SCC at 583 hours, test shutdown at 668 hours
- 40 mil crack noted by DE
- Is resolution between 18 and 40 mils?



No, 40 mils of SCC includes 85 hours of SCC growth

One question that routinely arises is what is the resolution of the EPD system for detecting SCC initiation. In this example, EPD indicated SCC initiation in an Alloy 600 samples stressed to 448 MPa and exposed to high purity water and 360°C (50 cc/kg H_2) at 583 hours, and the test was shutdown at 668 hours. Destructive examination of the specimen revealed a 40 mil deep crack. Other specimens that were similarly examined shortly after SCC initiation was detected by EPD were examined and determined to have ~18 - 40 mil deep cracks. This would suggest that the resolution of the EPD system is between 18 and 40 mils. In all cases, however, there was some SCC growth that was included in the measurement performed during destructive examination. Thus, the above crack depths require correction for crack growth.

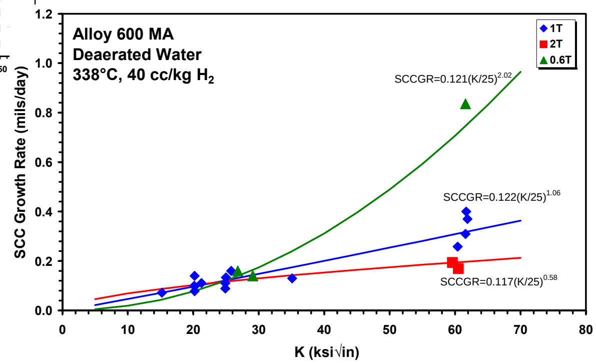
What is Resolution of EPD?



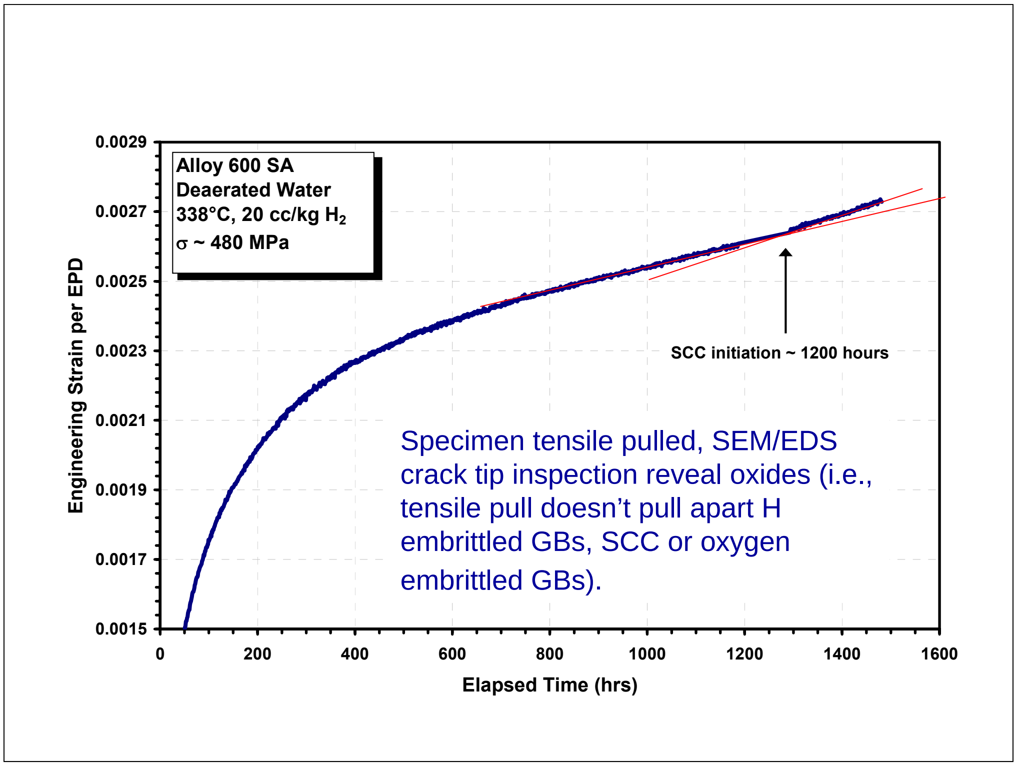
640°F Alloy 600 Heat NX5853G11 SCCGR as a function of the stress intensity factor (K) for various specimens sizes. Results suggest that at high K, SCCGR is enhanced by reduced specimen size.

CT and metallography indicate 40 mils crack extension

40 mils / (85 hours) = 11 mils/day



As stated previously, the crack depths measured during destructive examination include some finite amount of crack growth. An attempt was made to estimate the crack extension for the 85 hours (3.5 days) between the EPD detected initiation time and test shutdown. Prior testing of Alloy 600 under similar environmental conditions suggests that for a stress intensity factor (K) of 60 ksi√in, the SCC growth rate should be ~ 2 mils/day. However, the SCC growth rate verses K plot suggests that the SCC growth rate of button head tensile specimens could be greater than expected due to a “specimen size” effect. Specifically, at high stress intensity factors, higher SCC growth rate occur for compact tension specimens of smaller sizes, possibly due to a loss in constraint. An even greater SCC growth rate may occur in the button heat tensile specimens since these specimens are even smaller than 0.4T compact tension specimens at high K. In the unlikely event that EPD worked perfectly, all SCC crack growth would have occurred in the 3.5 days beyond “EPD indicated initiation”. In this case the resulting average crack growth rate for the maximum crack depth would be 40mils/3.5 days or 11 mils/day.



All specimens were inspected with a stereo-microscope at the end of testing. Most specimens were then tensile pulled ~50 mils (~4% strain) and inspected a second time with a stereo-microscope to characterize the SCC. The post-test tensile pull was done in order to In order to ensure that the post test tensile pull did not create or extend cracks in the specimens, one specimen that was tensile pulled was examined in the scanning electron microscope (SEM) with EDS and found to contain oxides at the crack tip. This suggests that tensile pulling does not pull apart hydrogen embrittled grain boundaries, SCC cracks, or oxygen embrittled grain boundaries.

Conclusions

- Active load testing coupled with the use of EPD for in-situ SCC initiation detection is an improvement over prior testing since:
  - the strain and strain history are accurately known and controlled
  - the data have less uncertainty than data derived from tests which rely on the period of time between specimen inspection intervals to estimate SCC initiation times
- Testing suggests that SCC initiation and SCC growth exhibit similar sensitivities to coolant hydrogen level.
- The thermal activation energy for SCC initiation in Alloy 600 is ~140 kJ/mol, which is very similar to the thermal activation energy measured for SCC growth. This value is much lower than many prior measurements, (e.g., 210-250 kJ/mol), likely due to coolant hydrogen effects.

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