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Comparison of Hydrogen Embrittlement Testing Methods of UNS N07718

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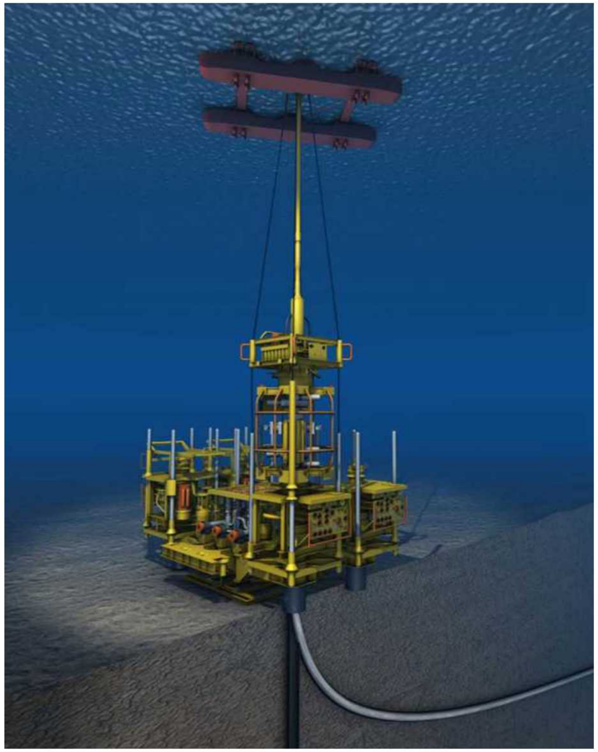
HE in Oil and Gas Environments

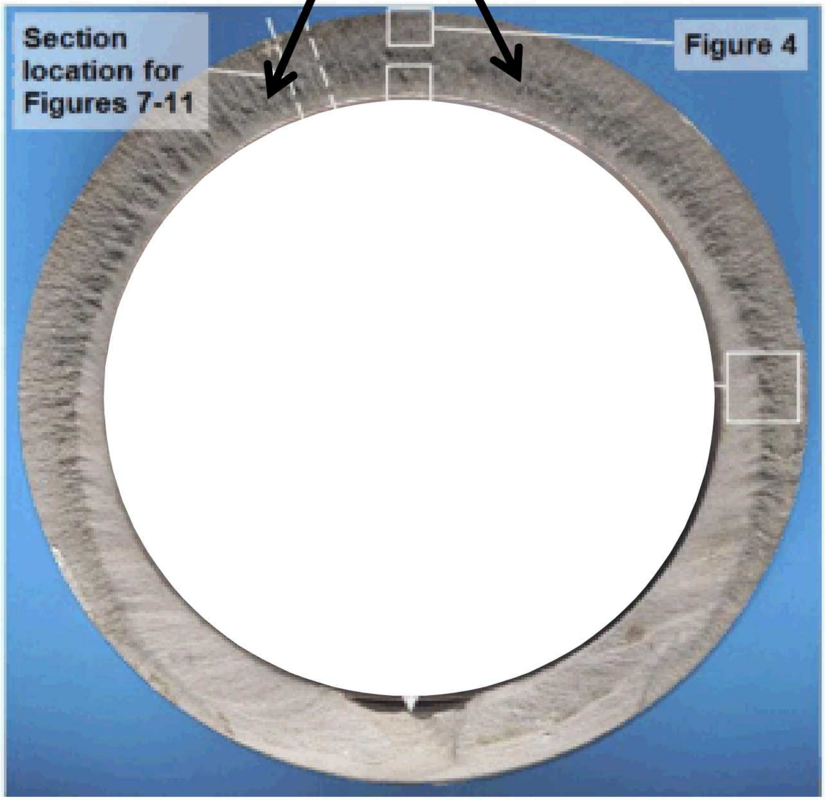
Failures of nickel-base corrosion resistant alloys (CRAs) can occur due to cathodic hydrogen embrittlement (HE)

Effects of γ” and γ’strengthening phases on HE are not well understood



http://www.afconsult.com/en/do-business/our-services/industry/oil--gas/subsea-engineering/

Hydrogen affected cracking region



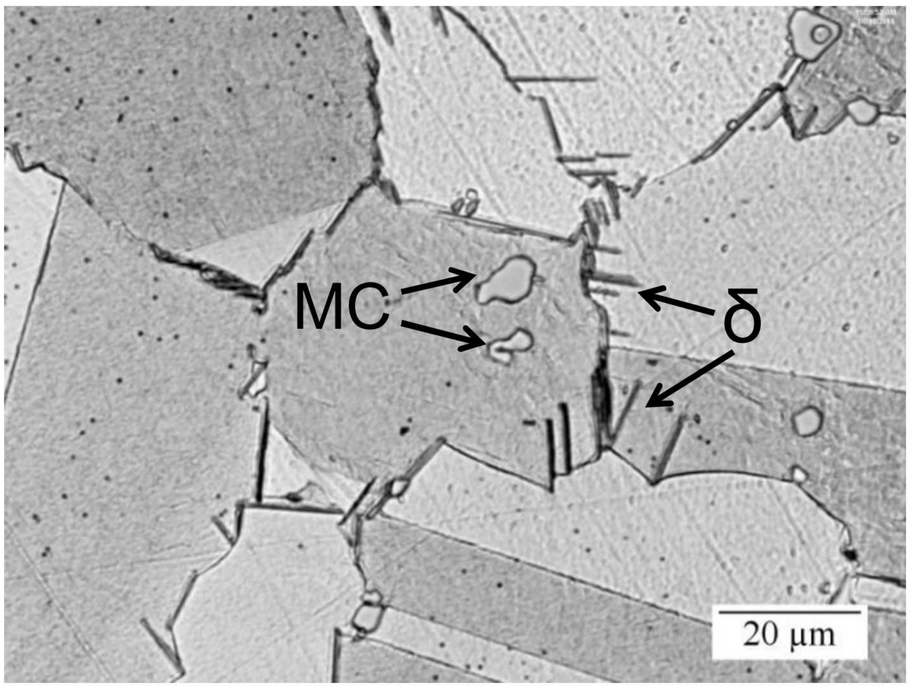
UNS N07716 Field Failure
Nice *et al.*, 2014

Alloy 718

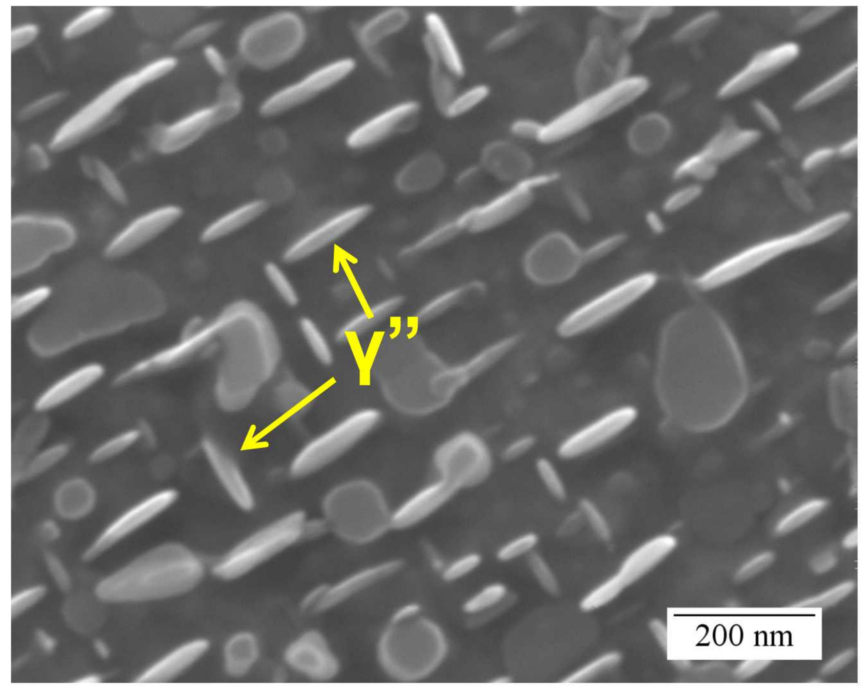
Alloy 718 Composition

wt pct	Ni	Cr	Nb	Mo	Ti	Al	Fe
Alloy 718	53.39	18.44	5.00	2.87	1.02	0.52	17.99

γ – Ni FCC Matrix γ'' ,γ' - Strengthening Precip. δ - GB Precip. MC



Solutionized at 1050 °C for 2.5 hrs and aged at 950 °C for 4 hrs then 800 °C for 8 hrs

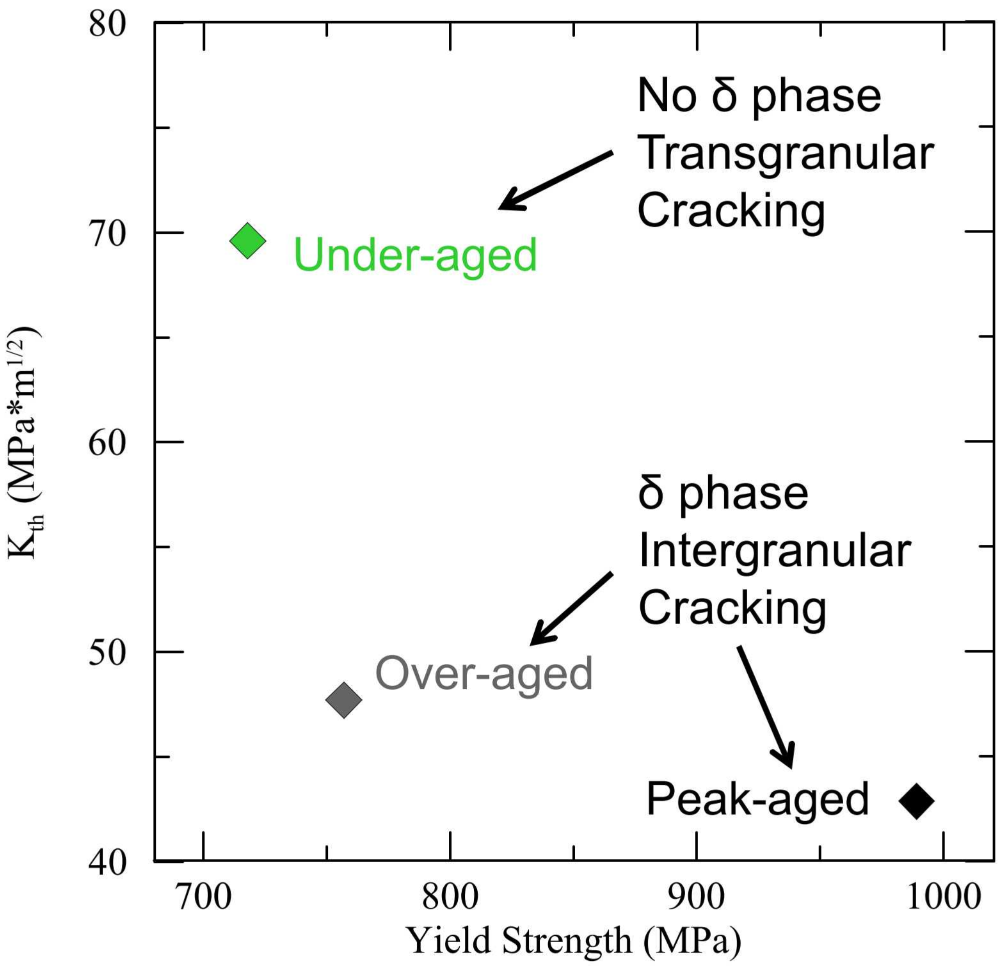


Solutionized at 1050 °C for 2.5 hrs and aged at 800 °C for 8 hrs

HE of Alloy 718

- Known to be susceptible to IGC when grain boundary δ phase is present
- HE susceptibility believed to increase with increasing yield strength

<u>**Are these observations dependent on test method?**</u>

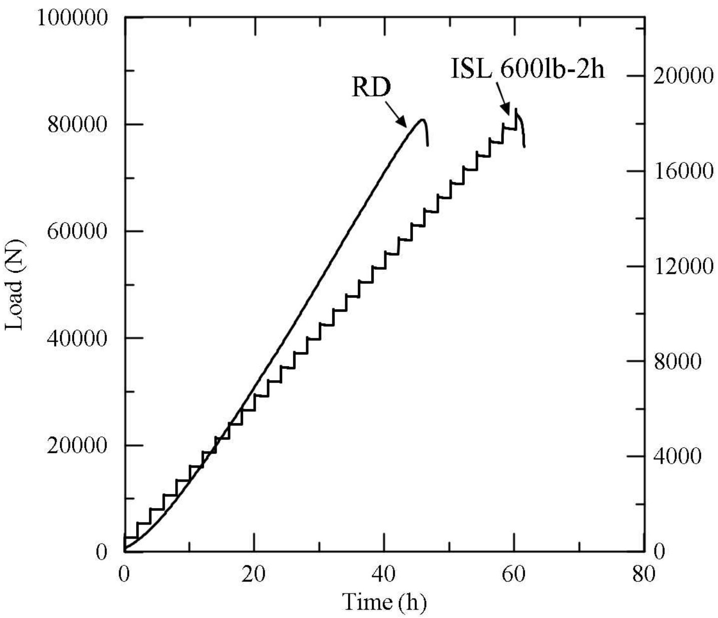


In situ HE Testing of Notched Specimens

Incremental Step Load (ISL):

- **Load is increased by 600 lbs every 2 h and then specimen is held at constant displacement**

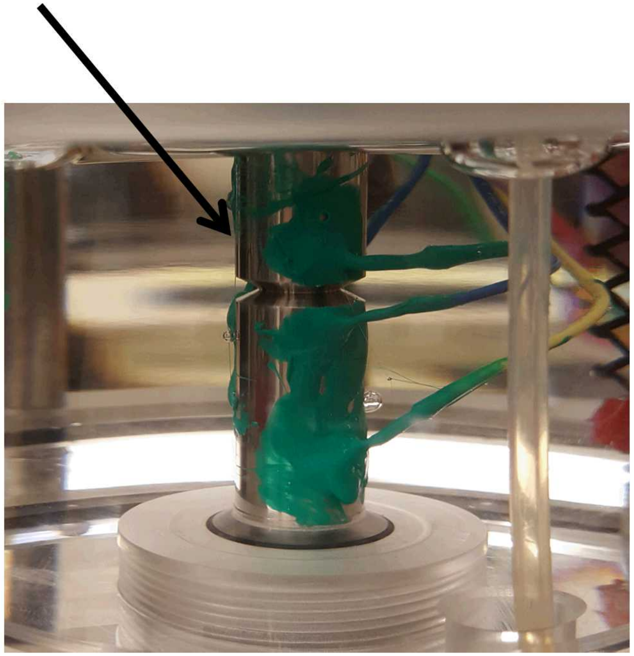
Rising Displacement (RD): continuously loaded throughout test



***In situ* electrochemical charging:**

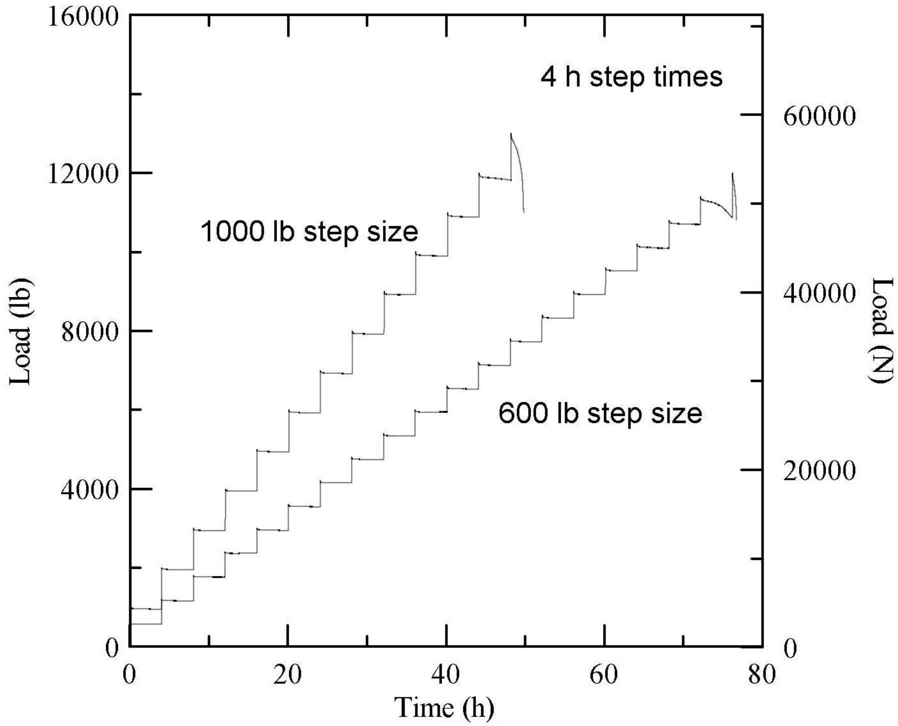
- **Solution: 0.5 M H_2SO_4**
- **5 mA/cm² current density**

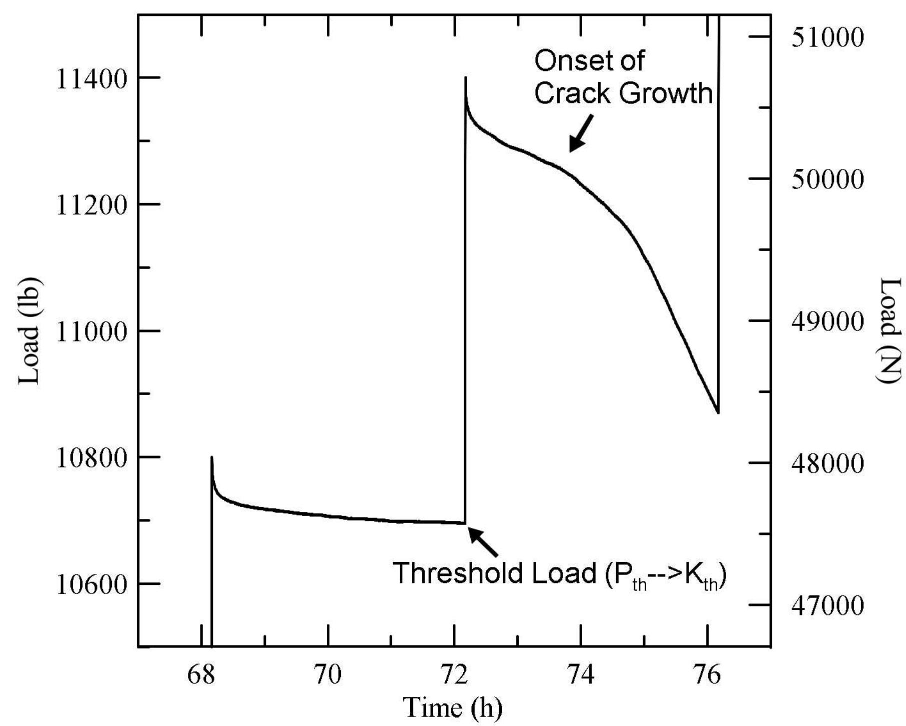
Circular notched tensile (CNT) specimens with sharp notch (K_t > 6)



ISL Test without DCPD

- Constant displacement test where the load is increased in steps after a pre-determined amount of time.
- Eventual decrease in load during a step indicates crack growth and determines K_{th} for tests without DCPD



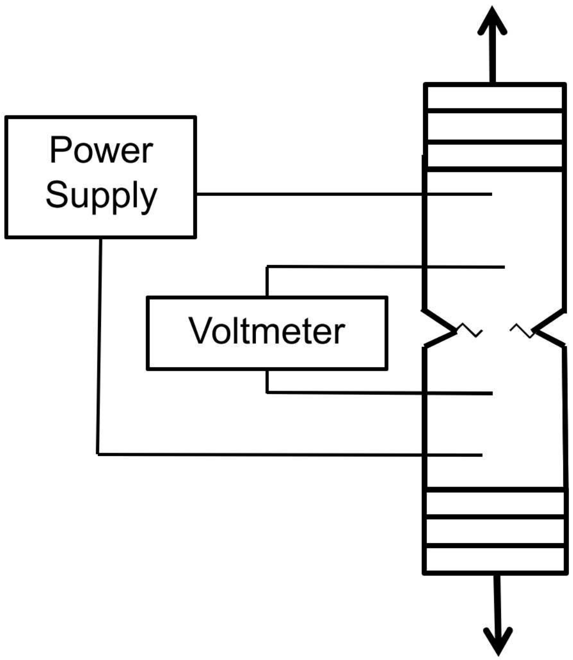


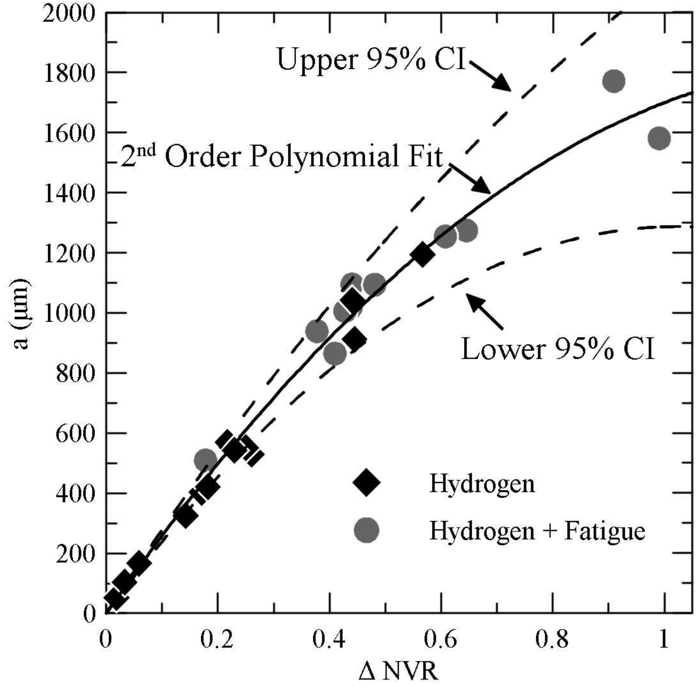
Direct Current Potential Difference (DCPD)

DCPD detects crack growth based on change in specimen resistance (R)

- **Constant current (I) applied across notch and voltage (V) measured**
- **Determine stress intensity factor for crack initiation (K_i) and onset of unstable crack growth (K_u)**
- **Produce crack growth resistance curves (K-R) (K vs crack length (a))**

$V = I * R$ ↑ **Crack length** → ↑ **Resistance** → ↑ **Voltage**

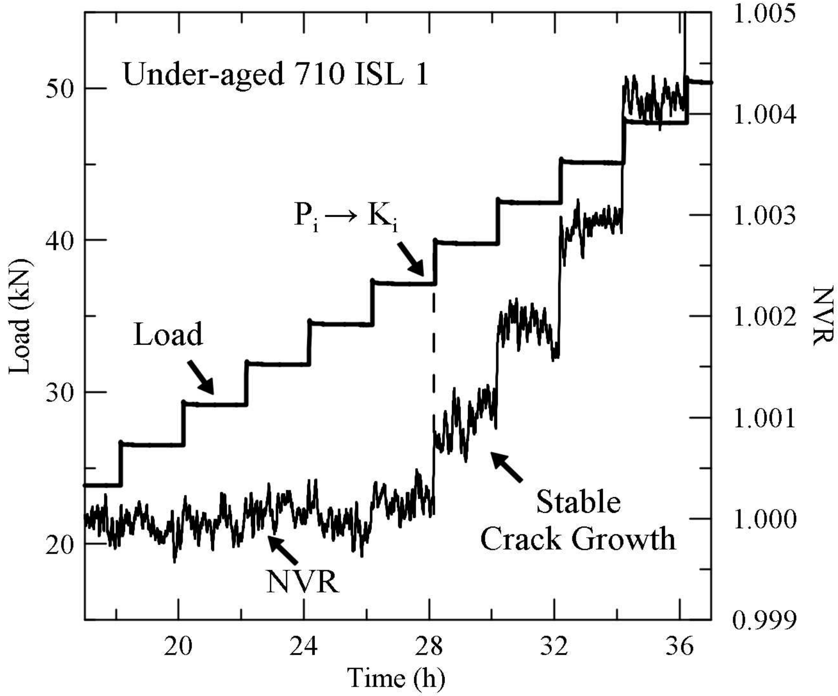




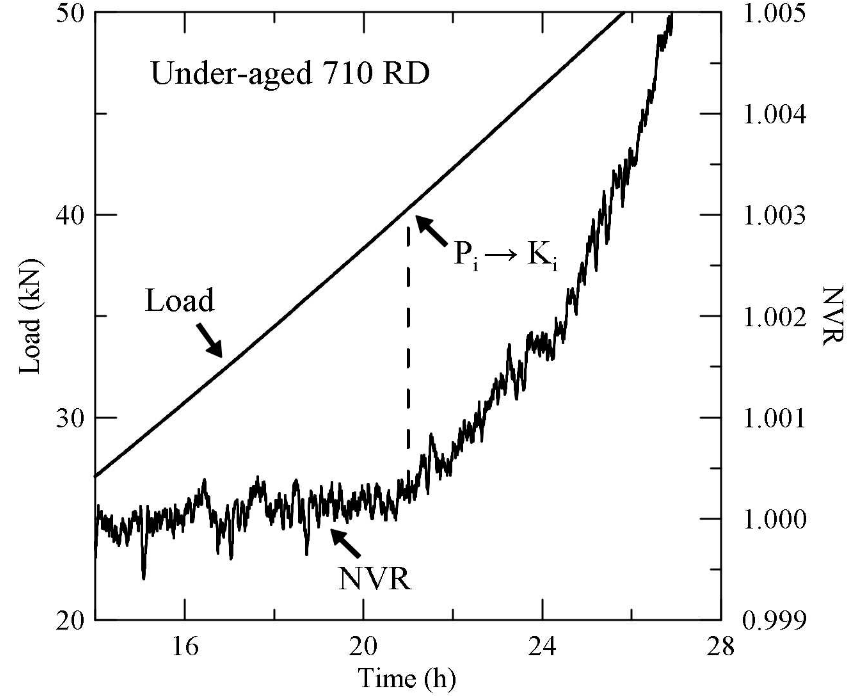
Notch voltage ratio vs crack length calibration curve

Crack Initiation (K_i)

ISL – Final load of step before stable crack growth



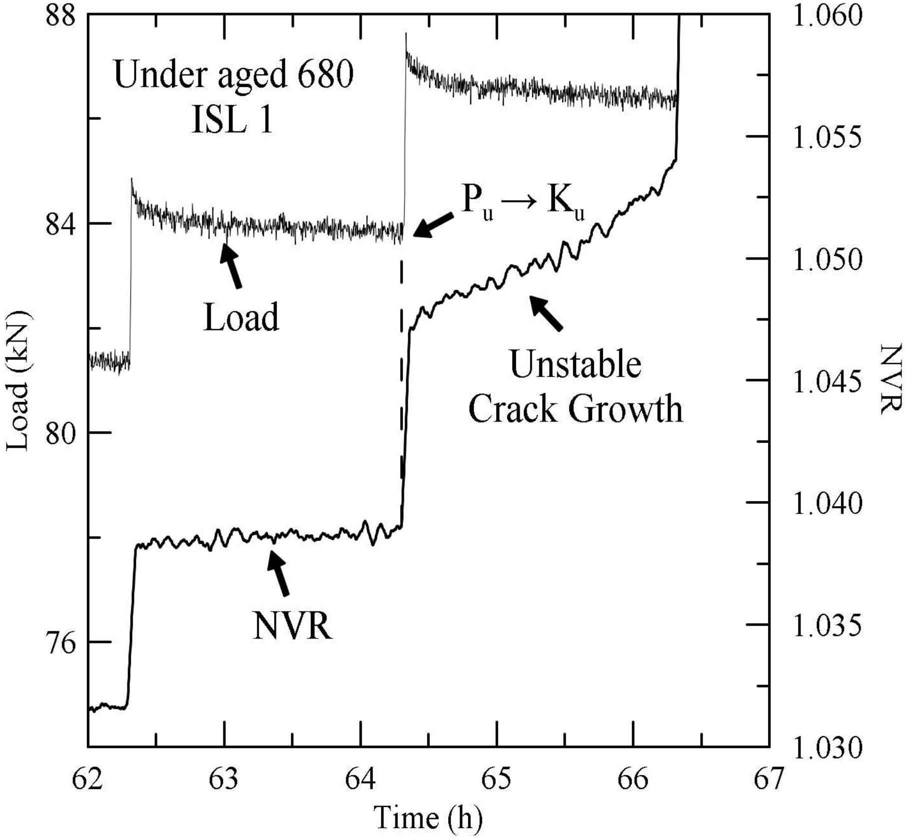
RD – Load at Voltage vs time inflection point



Unstable Crack Growth (K_u)

- **Onset of unstable crack growth represented by continuous increase in voltage ratio during a constant displacement hold**

- **No differentiation between stable and unstable crack growth in RD test**



SSR Experiments

- Smooth tensile specimens
- Strain rate of 1 x 10^{-6} s^{-1}
- Same environmental parameters as ISL and RD
- Three tests in hydrogen and three tests in air

$$\text{Total Elongation Ratio} = \frac{\text{Total Elongation in Hydrogen} \times 100}{\text{Total Elongation in Inert Environment}}$$

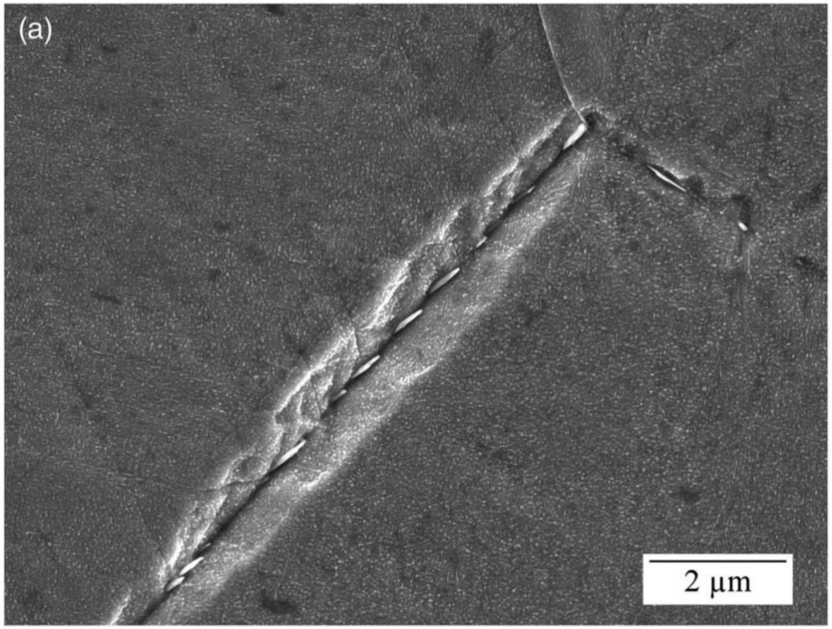
Increasing Total Elongation Ratio = Decreasing HE Susceptibility

Microstructures with Different Strength Levels and δ Phase

Microstructure	Aging Temp (°C)	Aging Time (h)	Yield Strength (MPa)	Total Elongation (%)
Under-aged 710	710	6.0	718	48
Double-aged	720 and 620	8.0 and 8.0	938	35
Under-aged 680	680	140	1094	27
Peak-aged	760	6	1095	30

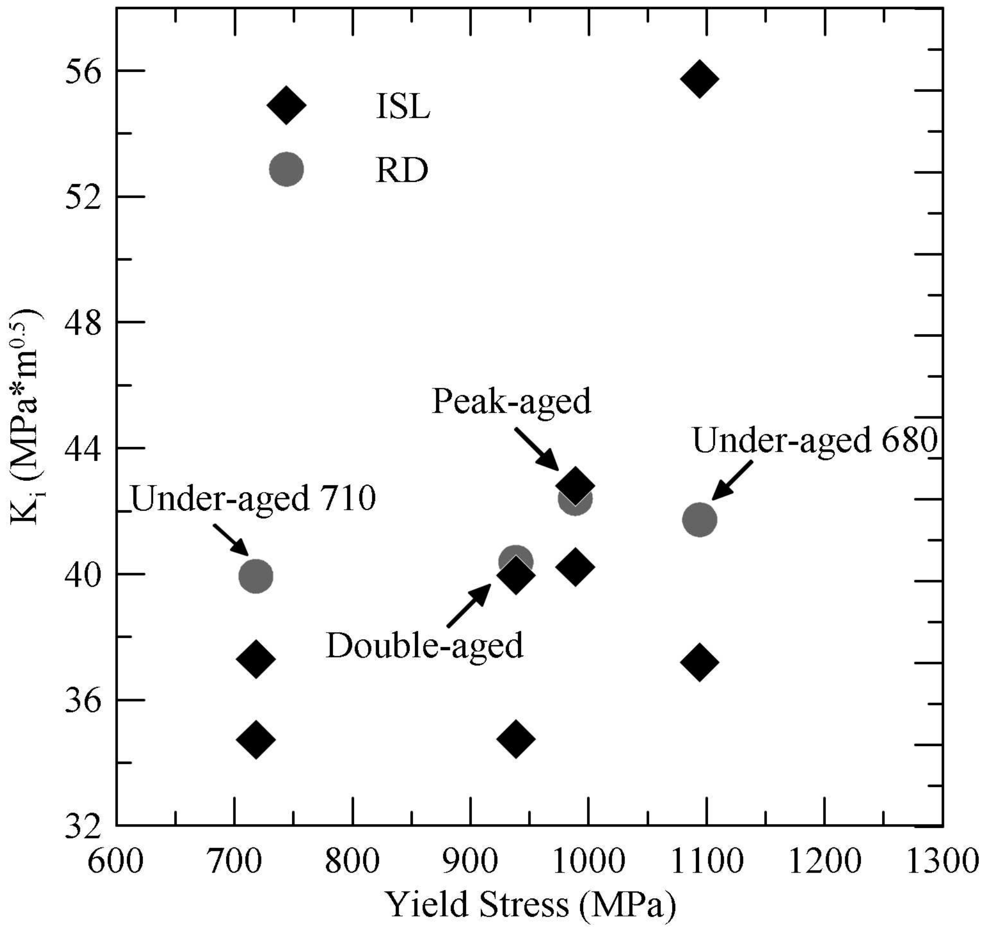
No δ phase in under-aged 710, double-aged, and under-aged 680

Small δ phase in peak-aged



δ phase in peak-aged

Minimal Effect of Microstructure on Crack Initiation (K_i)

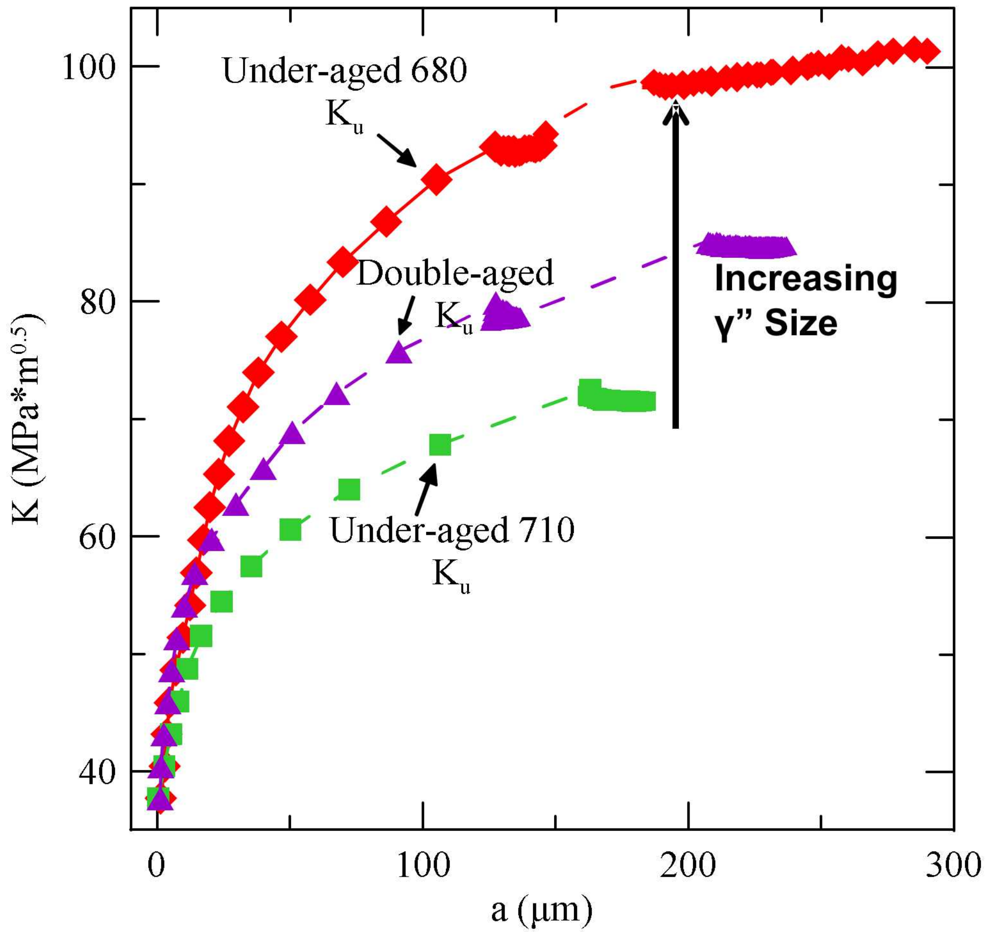


Minimal effect of microstructure on K_i

Similar K_i from RD for all microstructures

Large variance for under-aged 680

Increasing Under-aged γ" Size Increased HE Resistance



Higher K_u and crack growth resistance for under-aged 680, then double-aged, then under-aged 710

Same a_u for microstructures with no δ phase

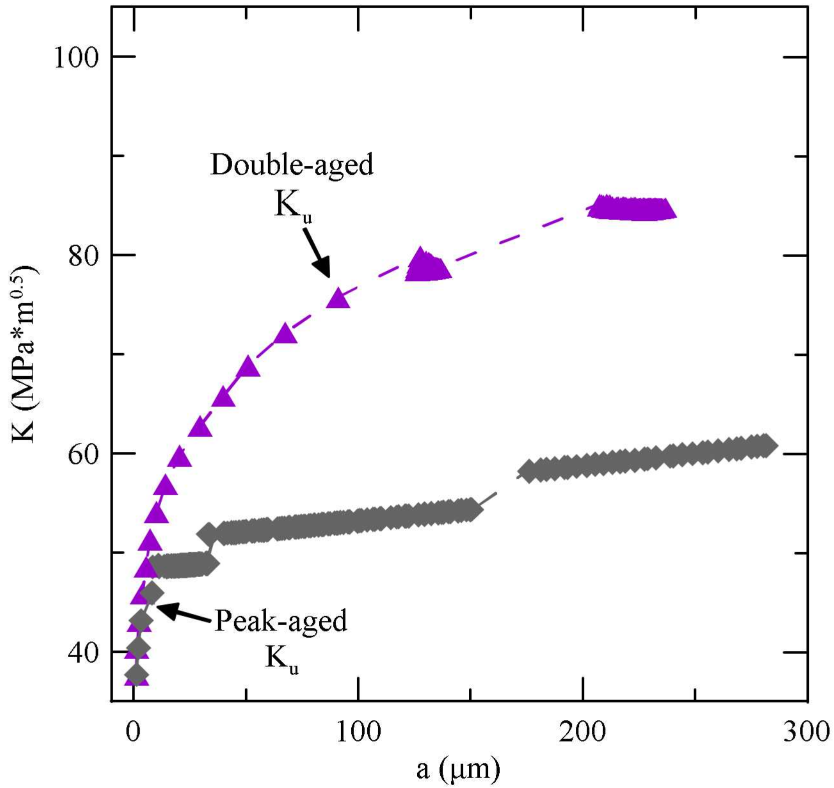
HE susceptibility decreased despite increase in yield strength for microstructures with no δ phase

δ Phase Decreases Crack Growth Resistance

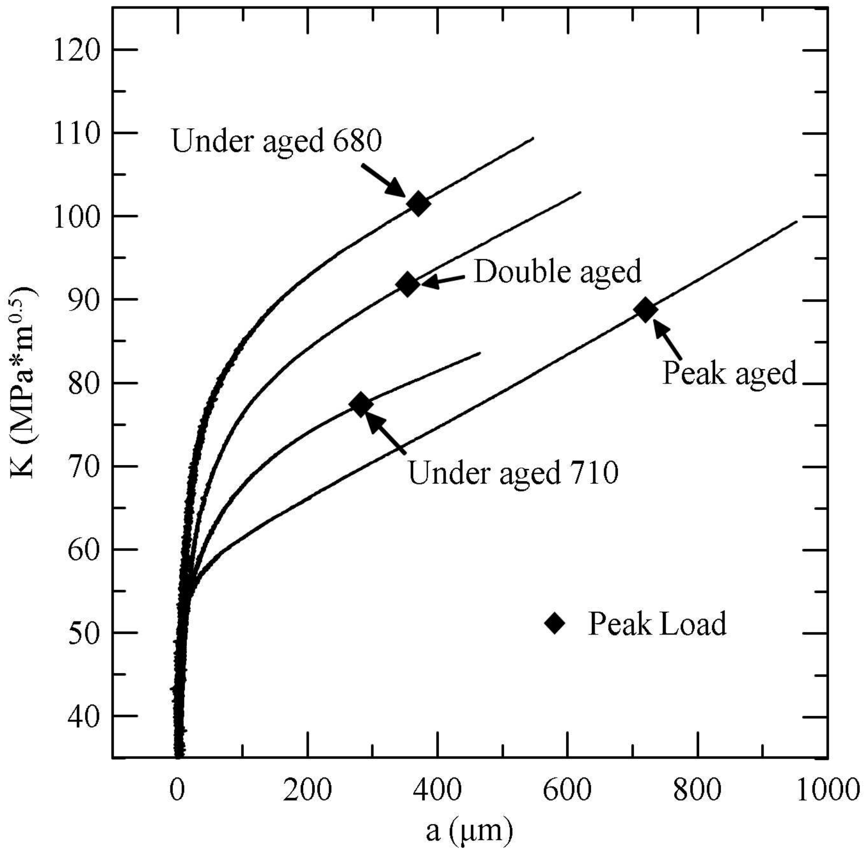
Peak-aged microstructure contains δ phase and has similar YS to double-aged

Much lower K_u but no change in K_i with δ phase

δ phase decreases amount of stable crack growth, but doesn't affect crack initiation



RD K-R Curves Match ISL

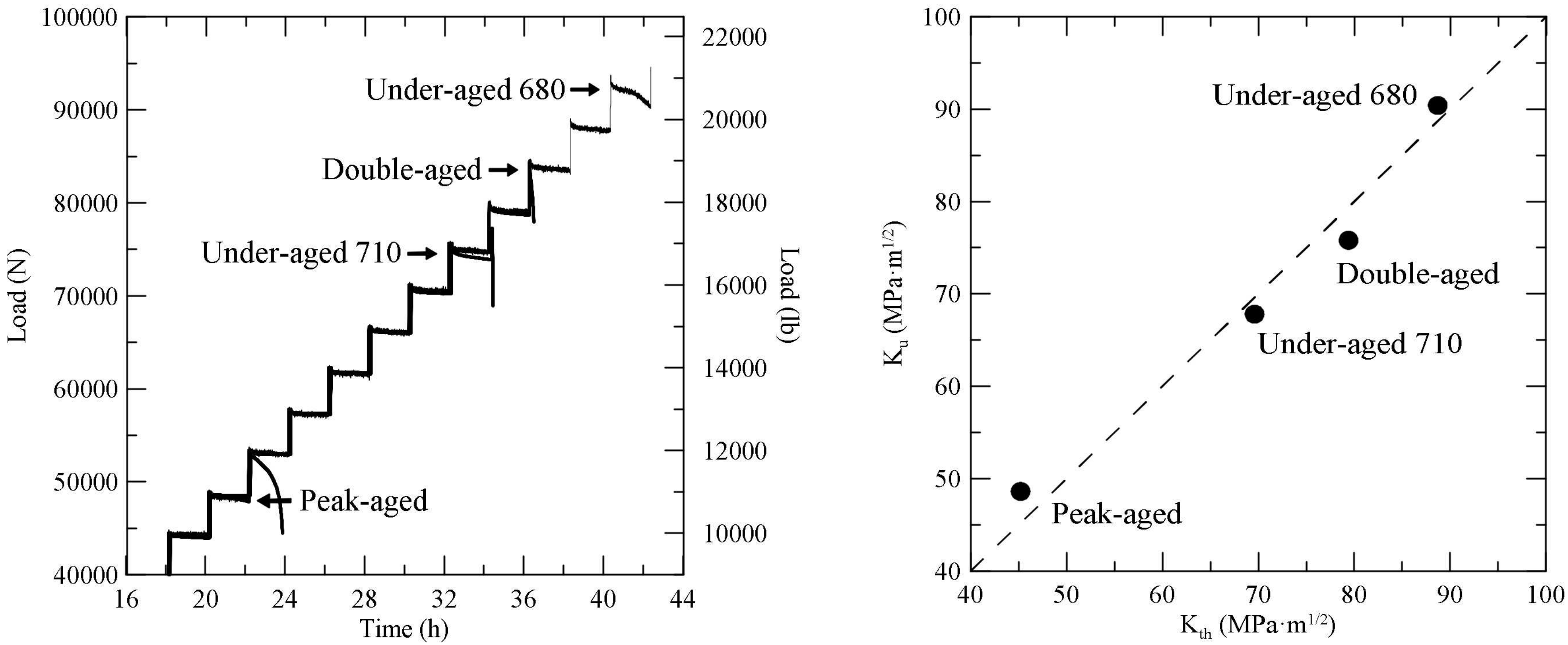


Similar trends from RD testing as to ISL testing

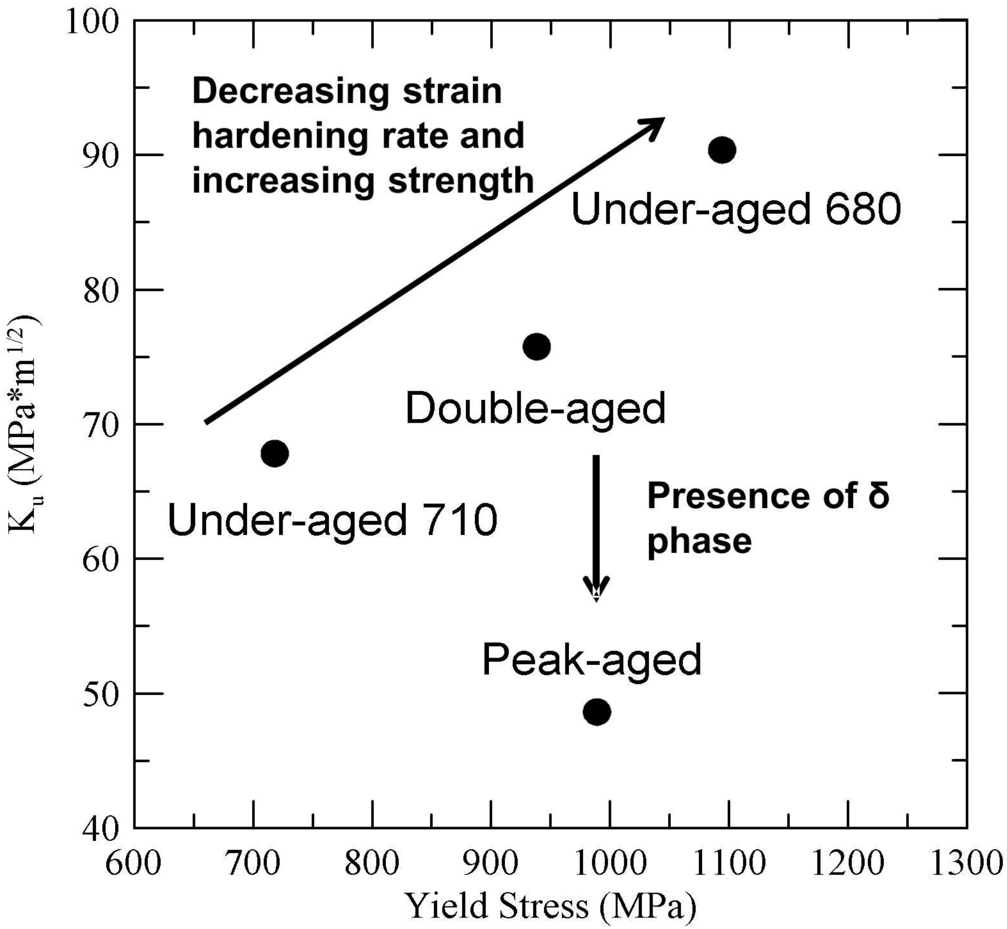
Microstructures with under-aged γ" show similar curve shape, but different curve shape for over-aged γ"

Similar Results for ISL with and without DCPD

K_{th} from ISL without DCPD does not detect crack initiation (Ki) but does gives similar result as K_u from ISL testing with DCPD



Greater Yield Strength Doesn't Increase HE Susceptibility

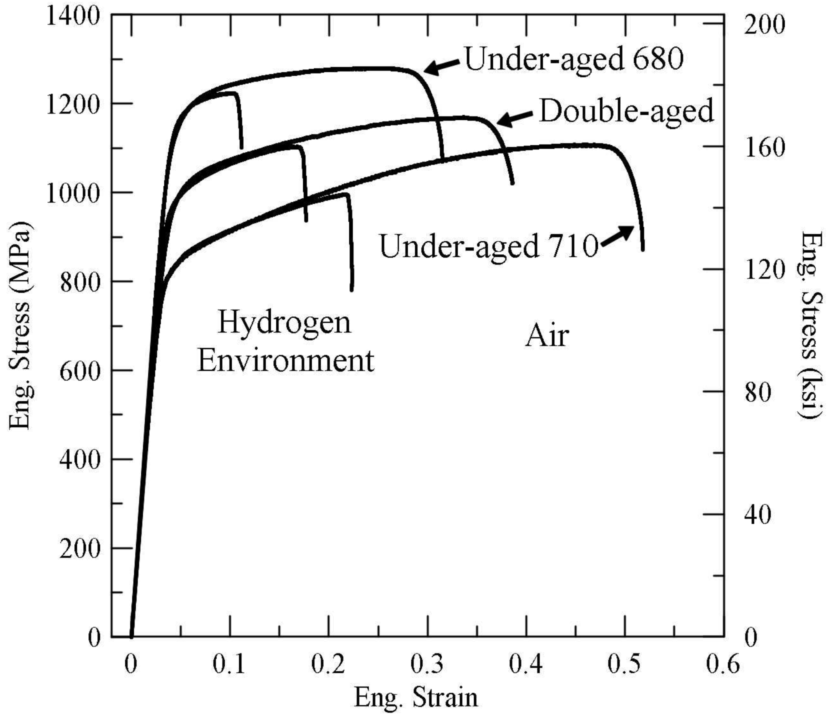


HE susceptibility decreased with decreasing strain hardening rate and increasing yield strength

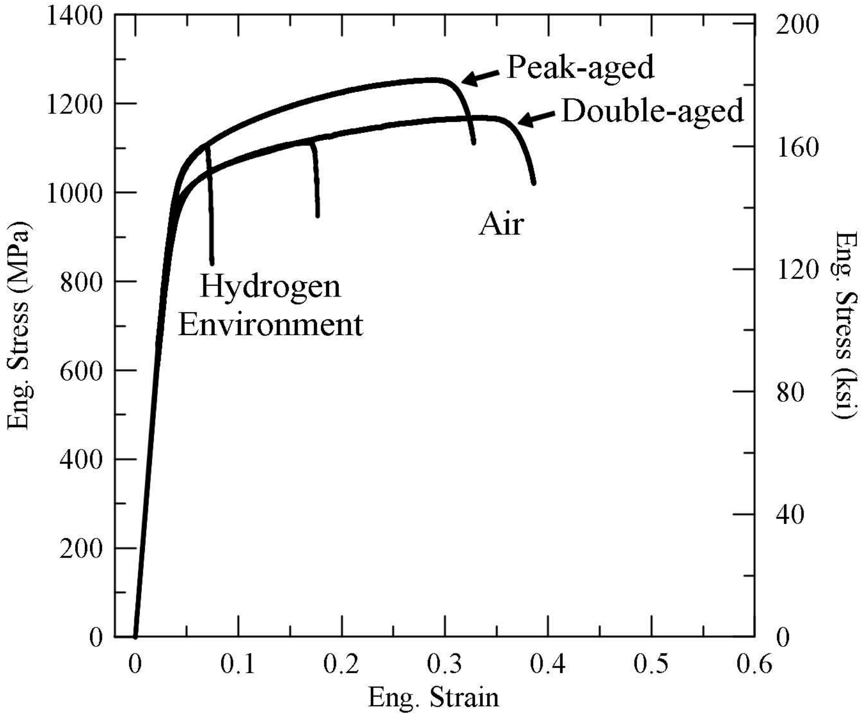
Small amounts of δ phase drastically increased HE susceptibility

SSR Results Don't Match ISL and RD Results

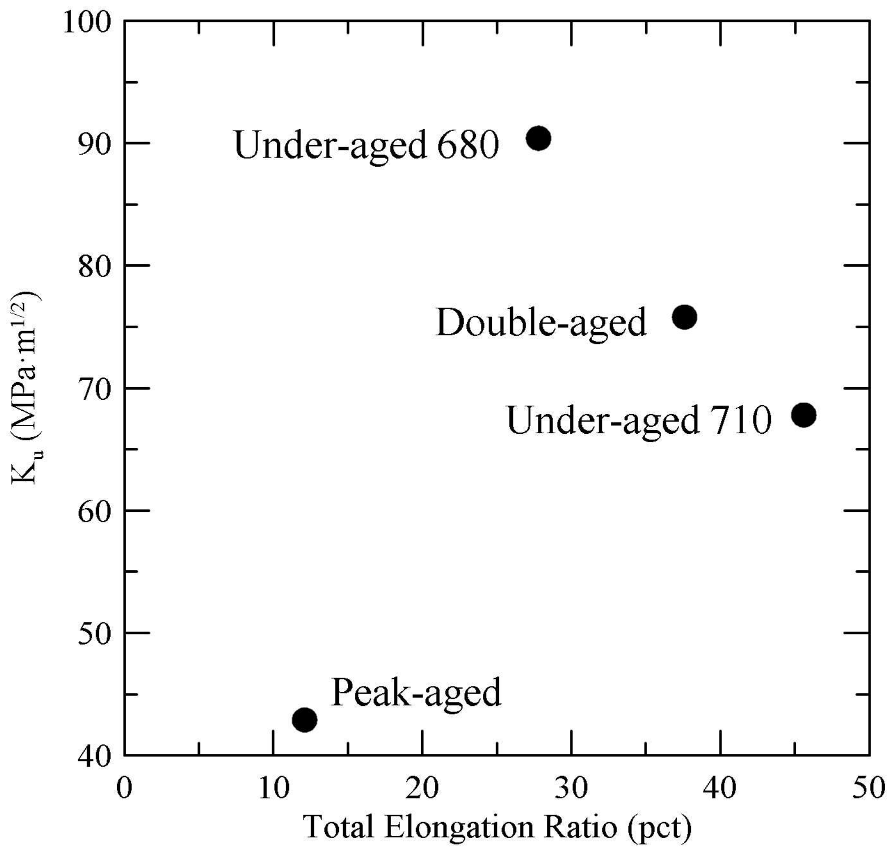
Higher strength microstructures are more susceptible in SSR test



δ phase still increases HE susceptibility in SSR test



ISL vs SSR Results

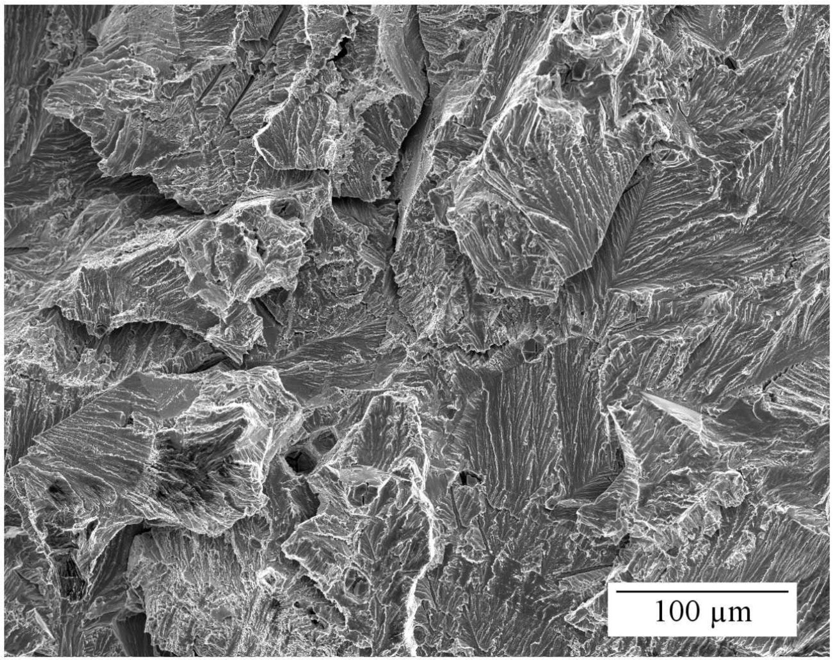


Microstructures with no δ phase exhibit inverse relationship between K_u in ISL test and total elongation ratio in SSR test

δ phase increases HE susceptibility in ISL, RD, and SSR tests

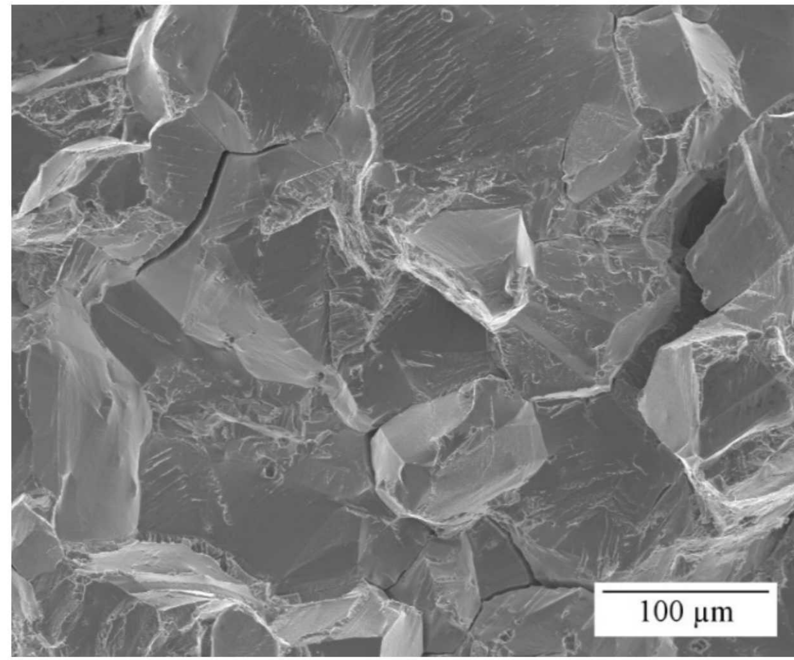
TGC with No δ, IGC with δ

Predominantly large faceted transgranular cleavage cracking (TGC) microstructures with no δ phase



Double-aged

Intergranular cracking (IGC) dominated for peak-aged microstructure containing δ phase



Peak-aged

Summary

- HE susceptibility decreased with increasing yield strength for microstructures with no δ phase
- δ phase reduced resistance to crack growth, but did not affect crack initiation
- ISL and RD testing with DCPD produced similar results
- ISL without DCPD did not detect crack initiation
- K_u from ISL testing exhibited an inverse relationship to total elongation ratio from SSR testing for microstructures with no δ

Thank you! Questions?