SAND2022-7591C

Sandia National Laboratories

Exceptional service in the national interest

Investigating the role of ferritic steel microstructure and strength in fracture resistance in high-pressure hydrogen gas

PVP2022-83915

Joe Ronevich[1], Brian Kagay[1], Chris San Marchi[1], Yiyu (Jason) Wang[2], Zhili Feng[2], Yanli Wang[2], Kip Findley[3]

[1]Sandia National Laboratories
[2]Oak Ridge National Laboratory
[3]Colorado School of Mines

ASME 2022 Pressure Vessels & Piping Conference, Las Vegas, NV

July 17-22nd, 2022

SAND2022-5612 V

U.S. DEPARTMENT OF ENERGY

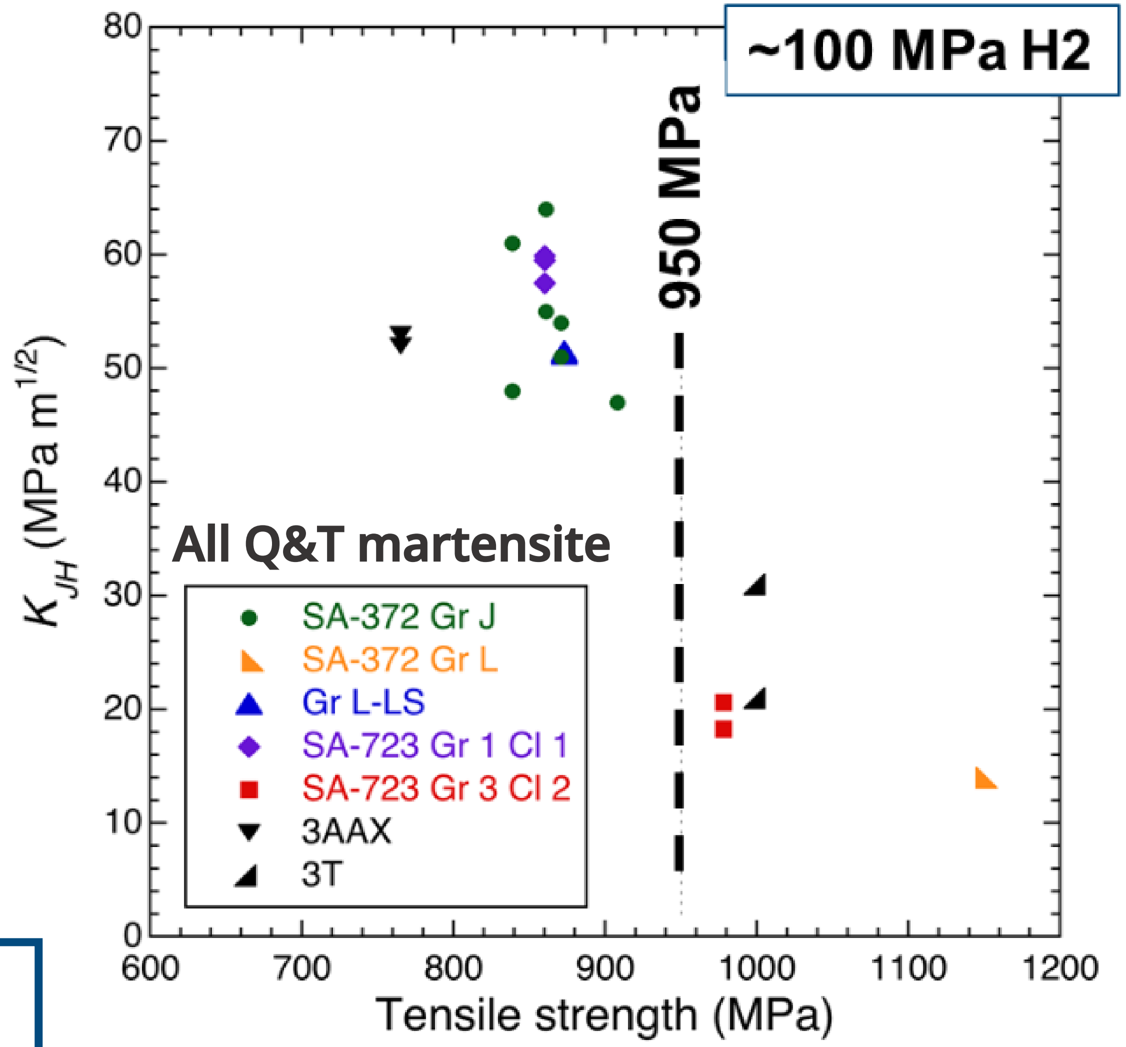
NNSA

Q&T martensitic steels in high strength condition have degraded fracture performance in hydrogen

Above ~950 MPa tensile strength:

→ Fracture resistance is < 30 MPa m$^{1/2}$

→ Limited data



Issue: In application, tensile strengths are often limited to less than 915 MPa (max allowed by ASME Code Case 2938)

→ Requires thicker walled vessels

Approach: Examine other high-strength ferritic-based microstructures to evaluate fracture performance in H_2

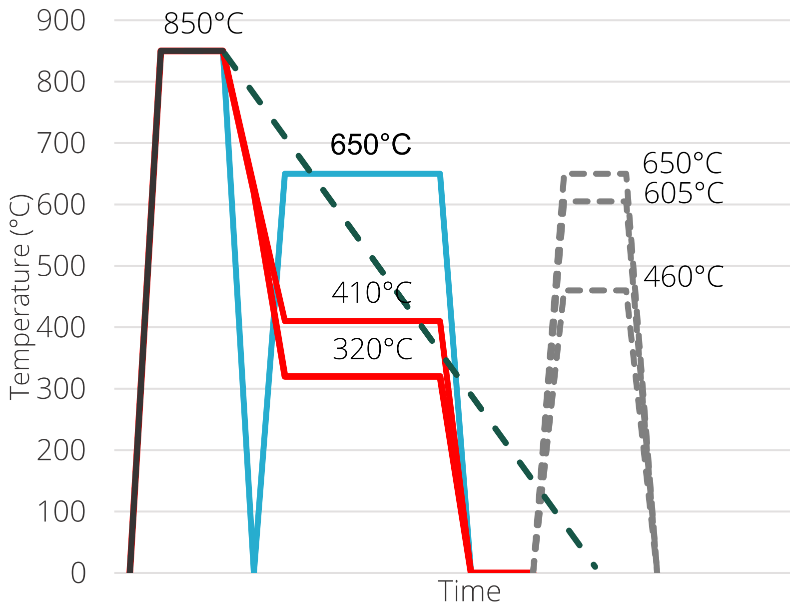
Heat treated a single 4340 to produce eight unique microstructures

4340 Chemical composition (wt. pct)

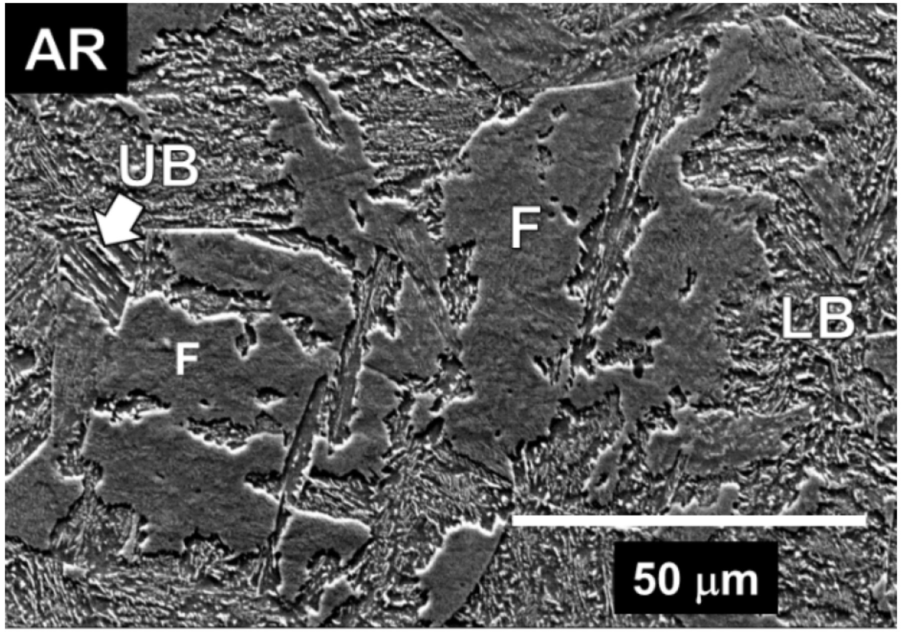
C	Mn	Si	Ni	Cr	Mo	Al	S	P	Cu
0.405	0.714	0.259	1.751	0.822	0.23	0.021	0.013	0.008	0.124

4340 steel:

- 44.5 mm diameter bar
- Hot-rolled with reduction ratio 22:1
- AR: Microstructure: ferrite, and upper/lower bainite



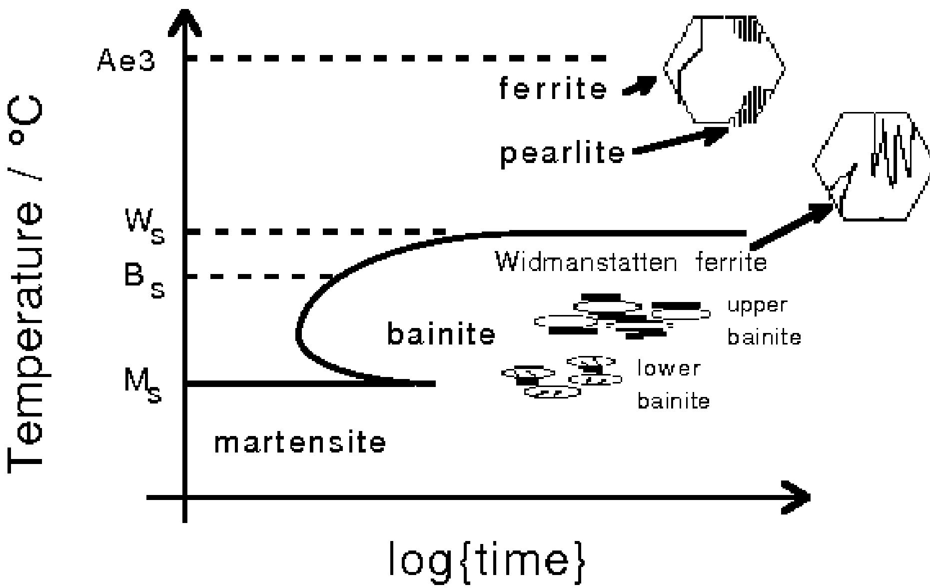
- All samples were normalized (850°C)
- 1 – Q&T 650°C
- 1 – Furnace cooled
- 6 – Austempered at
  - 410° (upper bainite)
  - 320° (lower bainite)
- Subsequent tempering to alter strength (460°C, 605°C, 650°C)



ELECTRON MICROGRAPH OF AS-RECEIVED 4340 STEEL. F=FERRITE, UB=UPPER BAINITE, LB=LOWER BAINITE.

Larger strength range, predominantly bainitic microstructures

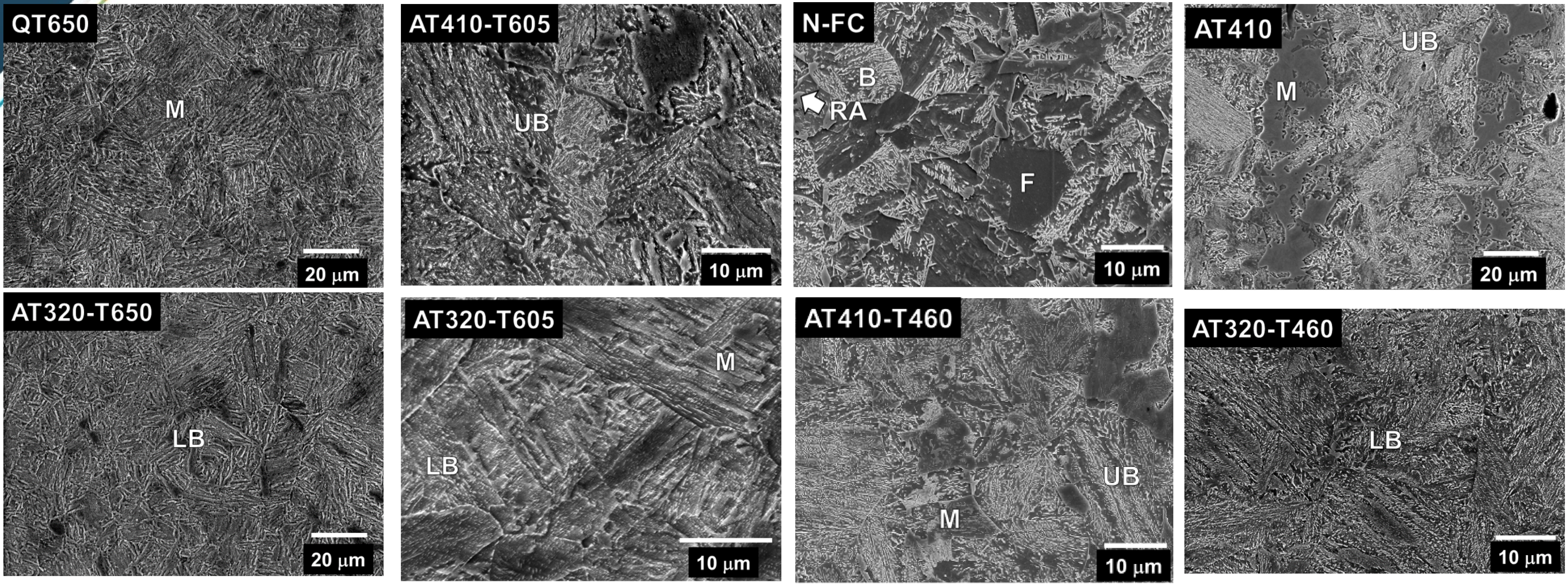
Sample	Heat treatment	Hardness (HRC)	YS (MPa)	UTS (MPa)	Phase
QT650	N-850°C-30min -WQ-T650°C-120min-WQ	28 ± 1	742	874	Tempered martensite
AT320-T650	N-850°C -30min-CC-AT320°C-60min-WQ-T-650°C-60min-WQ	28 ± 1	728	889	Tempered lower bainite
AT410-T605	N-850°C -30min-CC-AT410°C-60min-WQ-T-605°C-60min-WQ	28 ± 1	741	920	Tempered upper bainite, tempered martensite
AT320-T605	N-850°C -30min-CC-AT320°C-60min-WQ-T-605°C-60min-WQ	31 ± 1	859	992	Tempered lower bainite
AR	As-received	32 ± 1	721	1004	Ferrite, bainite
N-FC	N-850°C - 30min-FC	32 ± 2	721	1059	Ferrite, bainite, martensite, retained austenite
AT410-T460	N-850°C -30min-CC-AT410°C-60min-WQ-T-460°C-60min-WQ	33 ± 1	887	1100	Tempered upper bainite, tempered martensite
AT410	N-850°C - 30min-CC-AT-410°C -60min-WQ	34 ± 2	841	1195	Bainite, martensite
AT320-T460	N-850°C -30min-CC-AT320°C-60min-WQ-T-460°C-60min-WQ	40 ± 0.4	1158	1287	Tempered lower bainite



Ref: Bhadeshia, H. "Bainite in steels", Met Trans A, 1990

AR=as-received
AT=austempered
N=normalized
CC=continuous cooled
WQ=water quenched
T=tempered
FC=furnace cooled
QT=quenched & tempered

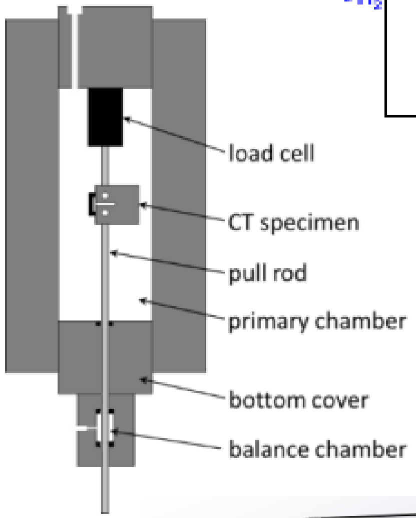
Decreased tempering temperature used to increase strength



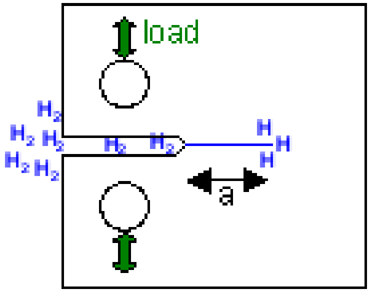
Increasing strength
Decreasing tempering temperature

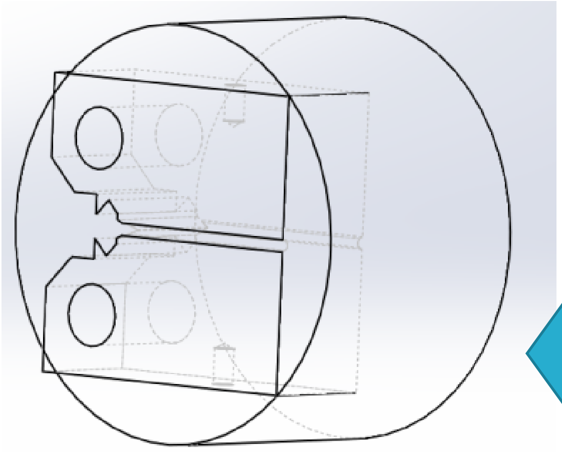
F = FERRITE, B = BAINITE, LB = LOWER BAINITE, UB = UPPER BAINITE, RA = RETAINED AUSTENITE, M = MARTENSITE.

Fracture tests performed in high-pressure gaseous H_2



Compact Tension
(C(T))





Instrumentation

- Internal load cell in feedback loop
- Clip gauge
- Direct Current Potential Difference (DCPD)

ASTM E1820 Elastic Plastic Fracture

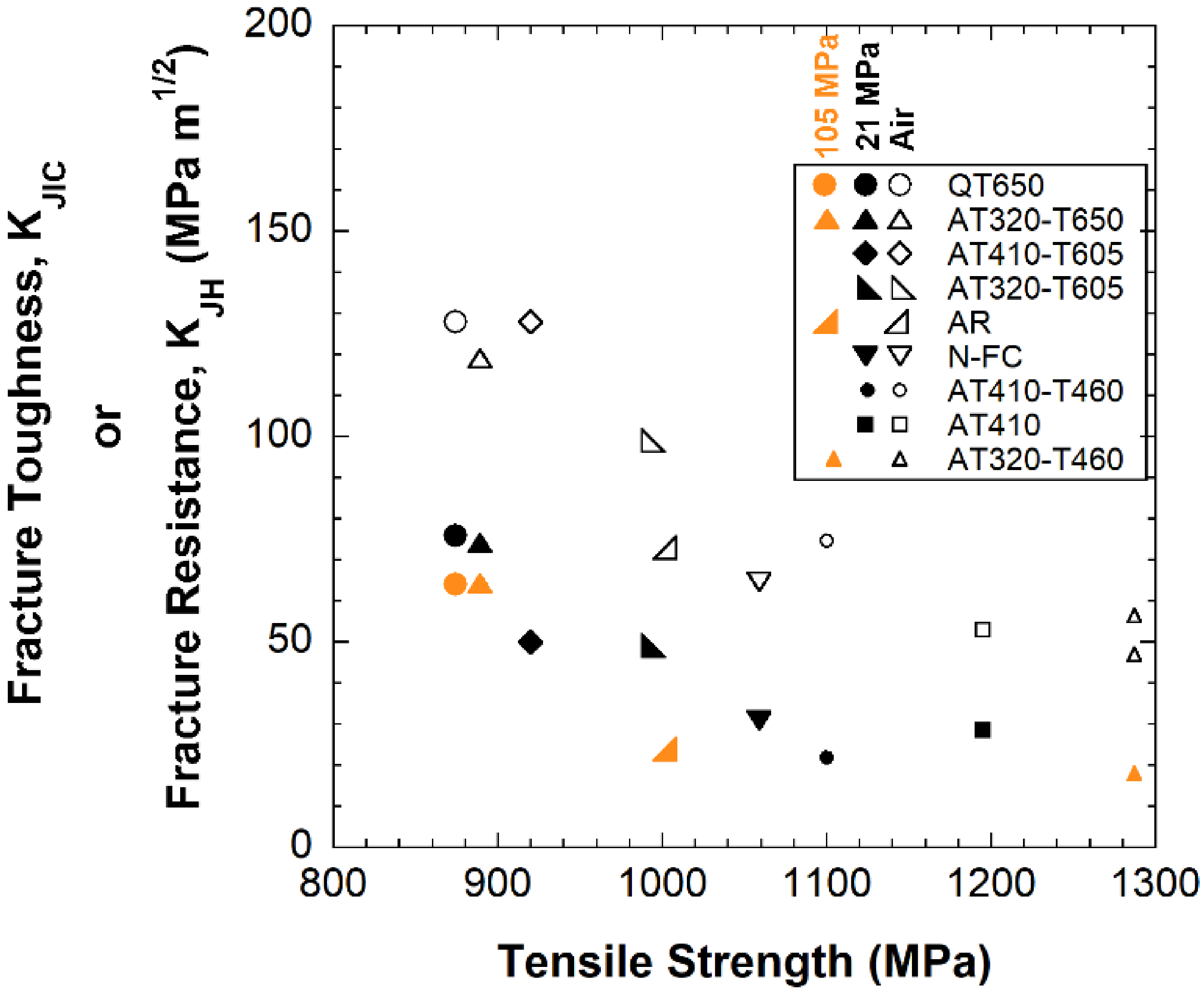
- $K_{JIC} = \sqrt{\frac{EJ_Q}{(1-\nu^2)}}$ Convert J to K
- Validity criteria
    - $B > 10(J_H/S_Y)$ and $b_o > 10(J_H/S_Y)$
- ***K_{JIC} or K_{JH} defined as intersection with 0.2 mm offset with J-R curve***

Environment

- Air
- 99.9999% H_2
    - 21 or 100 MPa pressure

C(T) sample extracted from bar so crack extends in radial direction

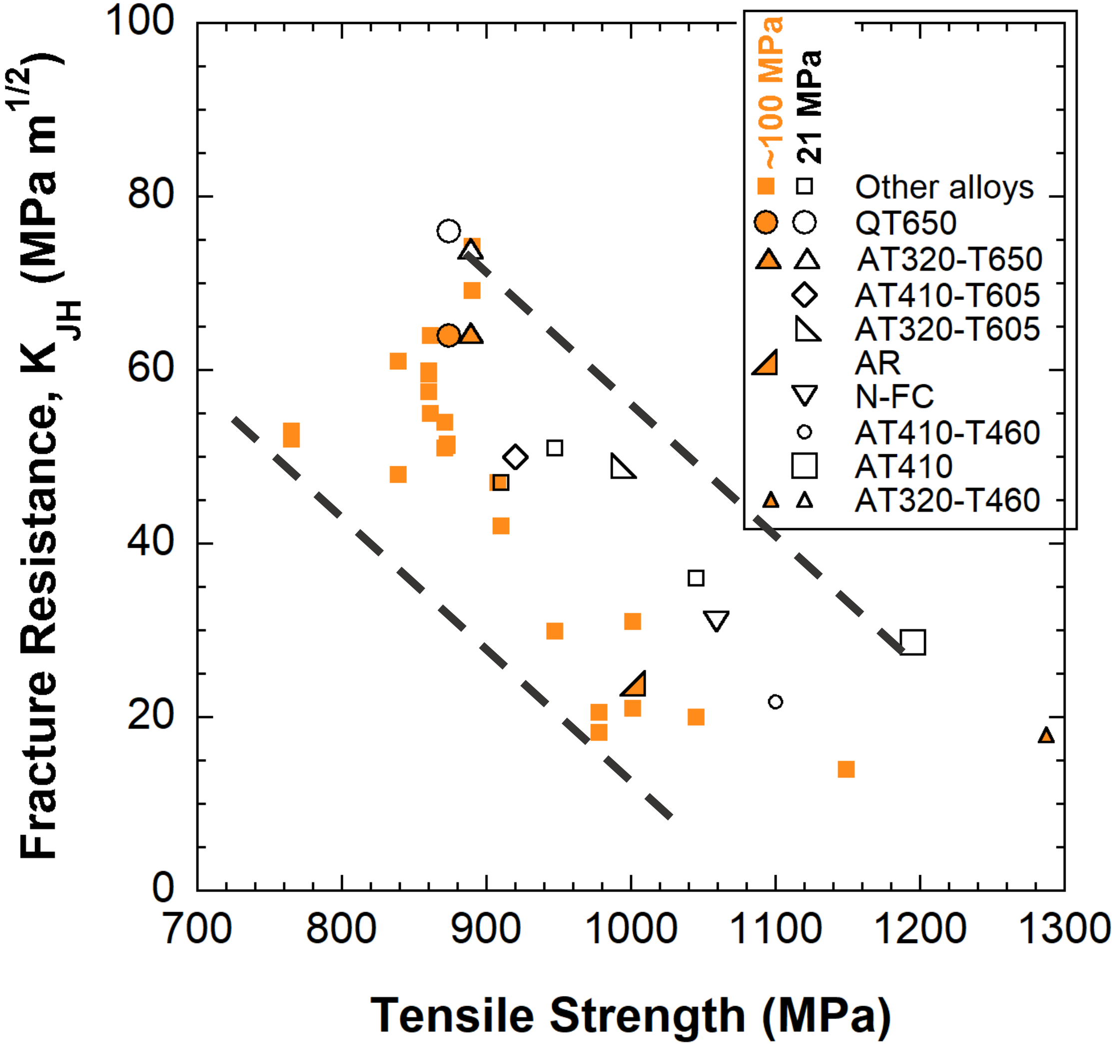
Fracture resistance of 4340 showed dependence on strength



What is role of:

- strength?
- microstructure?
- inclusions?
- pressure?

Strength has most dominant effect on fracture resistance in H_2

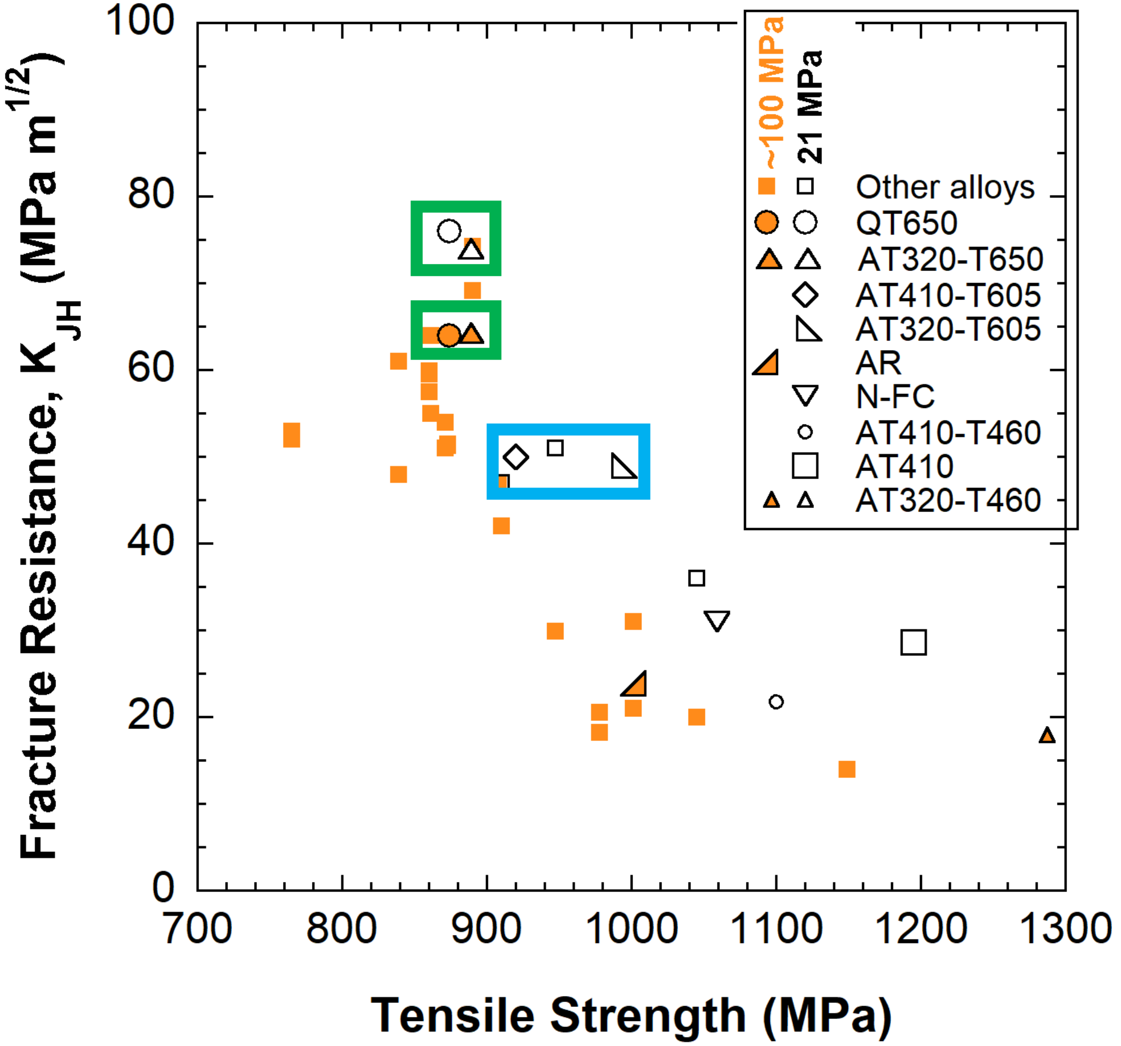


Despite differences in microstructures (Q&T martensite, upper bainite, lower bainite)

→ Increasing strength results in consistent decrease in fracture resistance in H_2

→ Other alloys consist of Q&T martensite

Microstructure played minor role in fracture resistance in H_2



Negligible effect:

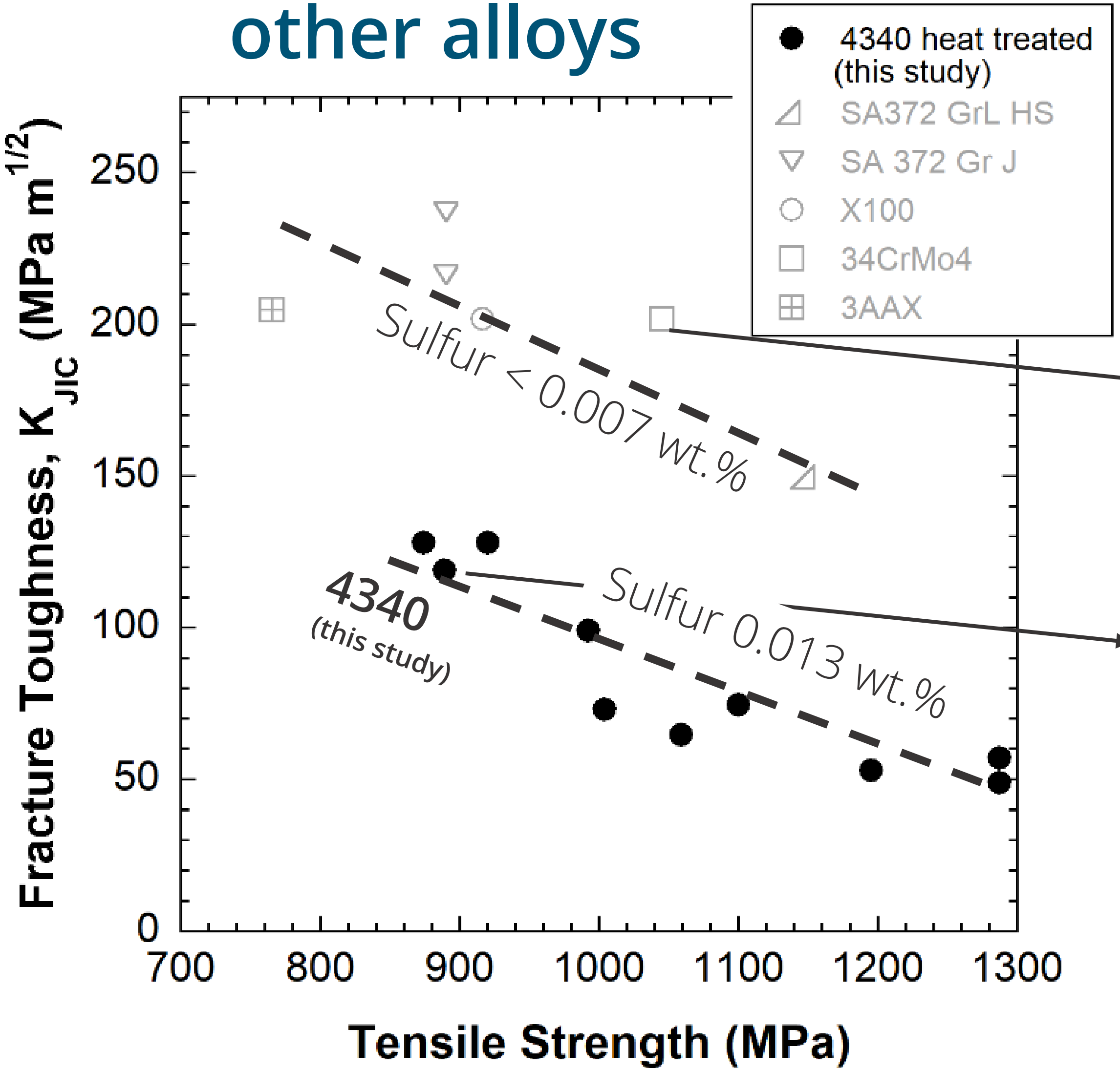
Similar K_{JH} for QT650 (Q&T martensite) & AT320-T650 (lower bainite) at both 21 and 100 MPa H_2

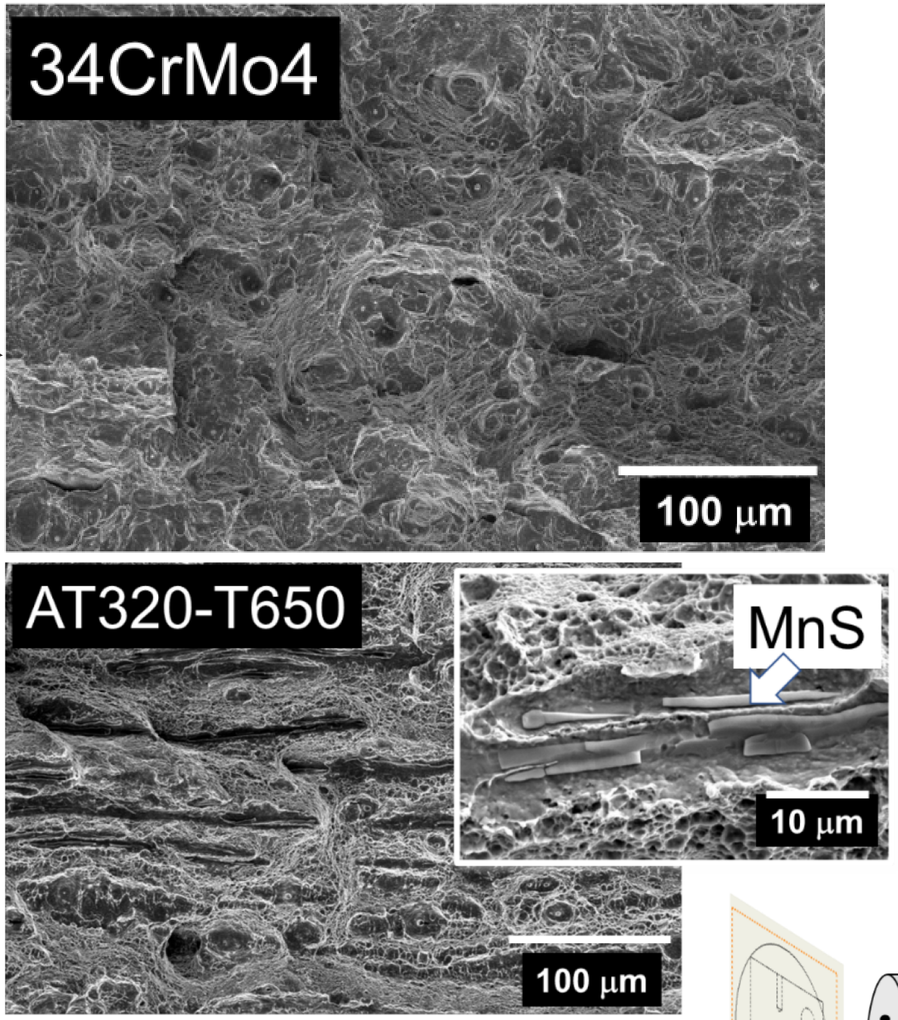
Small effect

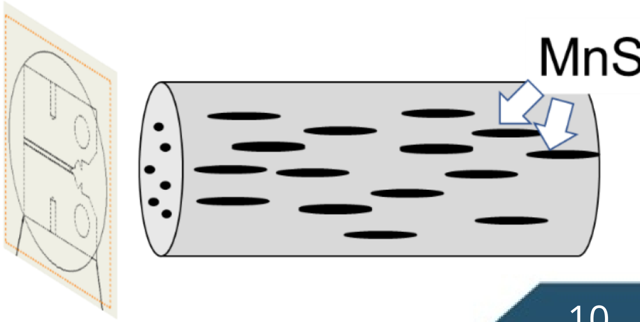
Similar K_{JH} for AT410-T605 (upper bainite) AT320-T605 (lower bainite) and a Q&T martensite (other alloys) at 21 H_2 but strength is higher for lower bainite

Lower bainite has small improvement over upper bainite

Fracture toughness in air of 4340 was 30-50% lower than other alloys

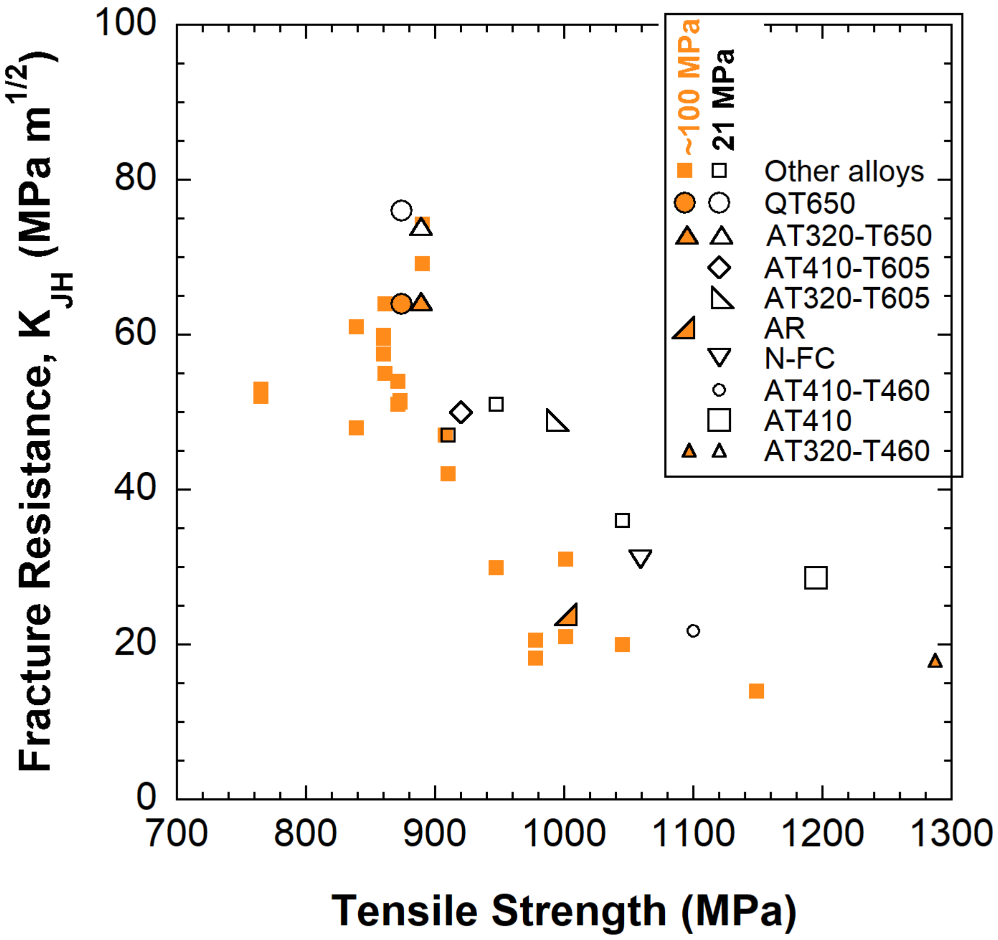




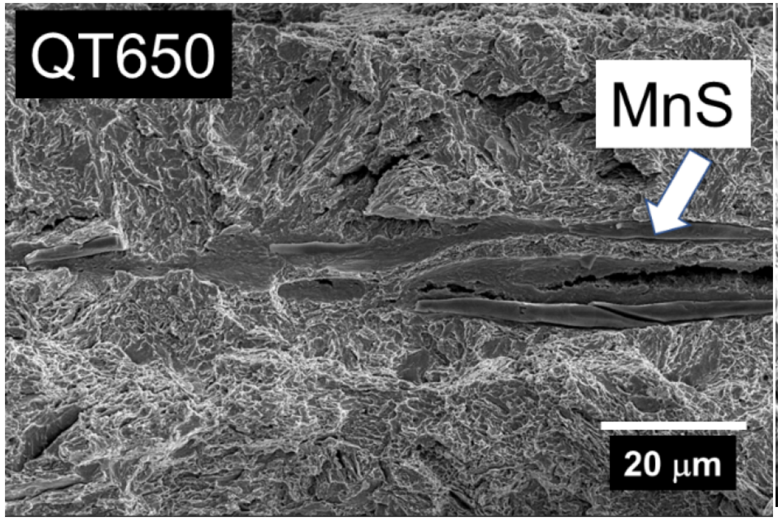


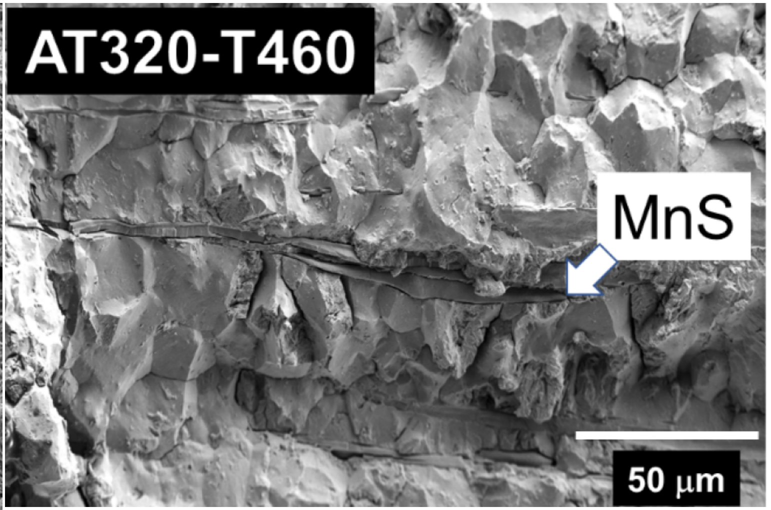
Higher MnS inclusion content resulted in reduced fracture toughness in air

MnS inclusions did not affect fracture resistance in H_2



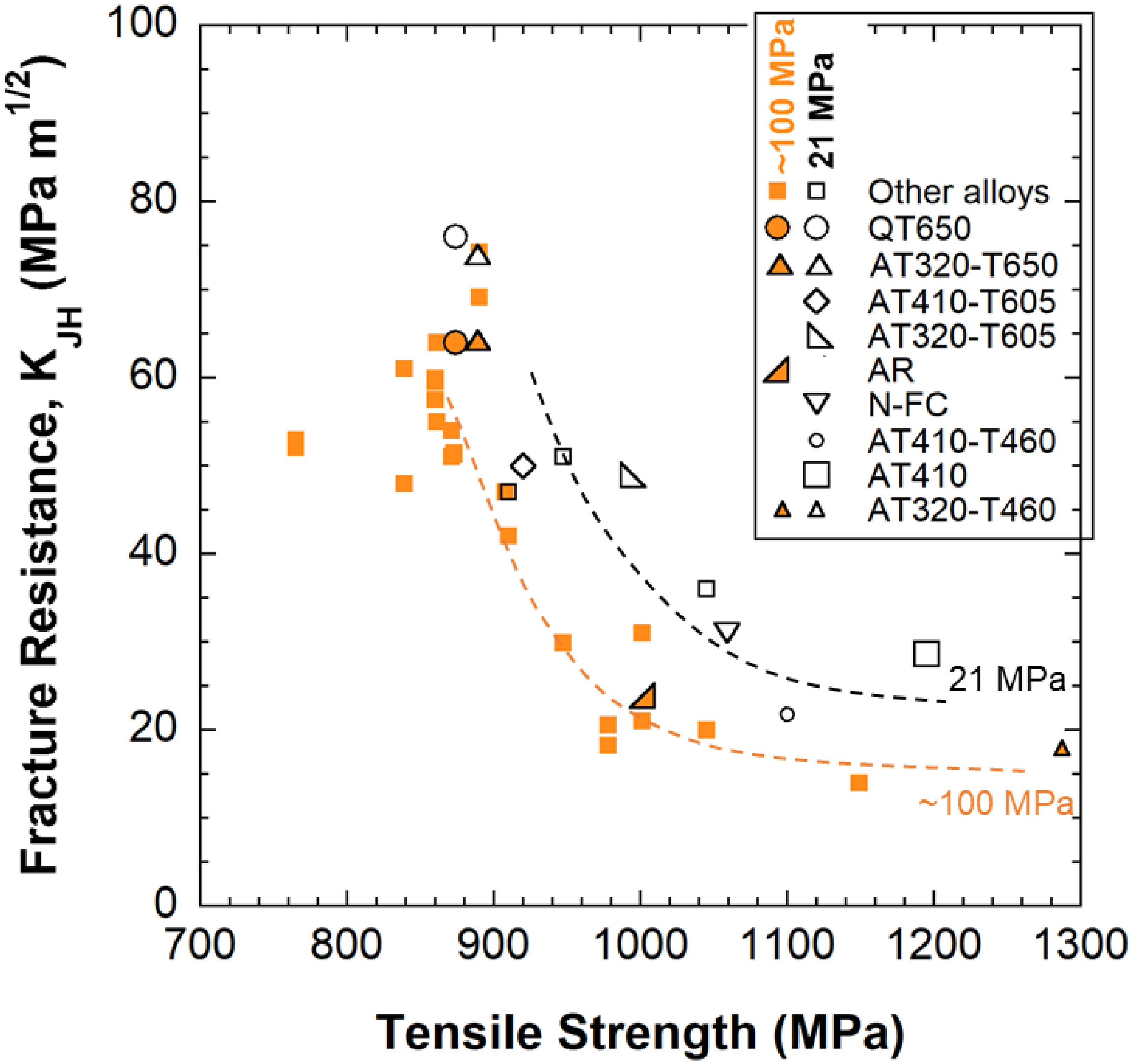
→ Fracture resistance values in gaseous H_2 were comparable to other (low sulfur alloys)





MnS inclusions do not participate in fracture when ductile void coalescence is absent

Pressure had only a small reduction in fracture resistance in H_2



Pressures of 21 and 100 MPa were tested for 4340 and other alloys

→ 5 to 20 MPa m$^{1/2}$ decrease in fracture resistance was measured due to pressure

→ Fracture degradation from air to 21 MPa H_2 was more pronounced than from 21→ 100 MPa H_2

Pressure and fracture resistance have a non-linear relationship

Summary

- Nine unique microstructures were developed from 4340 bar and fracture tested in hydrogen gas
  - **Strength**: Most dominant influence on fracture resistance in H_2
  - **Microstructure**: Minor influence (lower bainite performed better than upper bainite)
  - **Inclusions**: MnS inclusions affected fracture in air when damage mechanism was ductile void coalescence, negligible effect in H_2
  - **Pressure**: Non-linear effect due to pressure comparing 21 and 100 MPa H_2
- Fracture toughness in air is not a good indicator of performance in H_2
  → Hydrogen is the great equalizer

Thank you for your attention!

Joe Ronevich

jaronev@sandia.gov

Acknowledgements: We would like to acknowledge the help of the Hydrogen Effects on Materials Lab team consisting of James McNair, Brendan Davis, and Jeff Campbell. This work is supported by the U.S. Department of Energy, through the Office of Energy Efficiency and Renewable Energy's (EERE) Hydrogen and Fuel Cell Technologies Office (HFTO). ***Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.***