Small Specimen Mechanical Property Testing of Irradiated Materials

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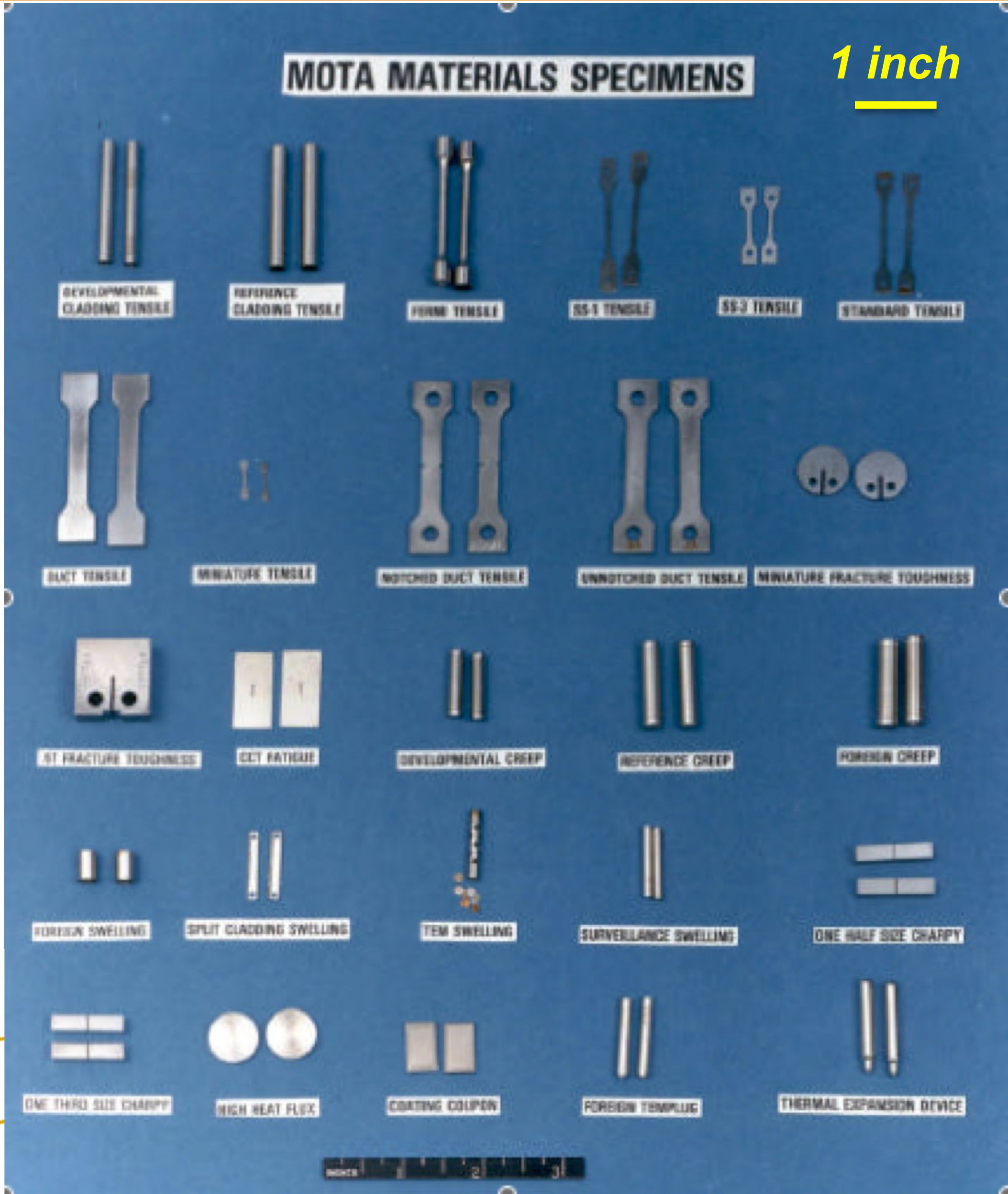
Overview of Mechanical Property Measurements from Small Specimens

- Long history (30+ years) of small specimen testing in support of characterizing irradiated materials for fusion, fission, spallation neutron irradiation studies, and more.
- Necessity typically based on limited material, irradiation volume, or post-exposure radioactivity.
- Miniaturized versions of standard test specimen geometries:
  - ***Tensile testing***
  - Compression testing
  - ***J-integral fracture toughness***
  - Charpy impact
  - Fatigue testing
- Some small specimen tests using non-standard geometries:
  - ***Shear punch to estimate tensile properties***
  - Ring pull
  - ***Automated microhardness***
  - Ball punch testing to estimate toughness
  - Pressurized tube in-reactor creep and thermal creep

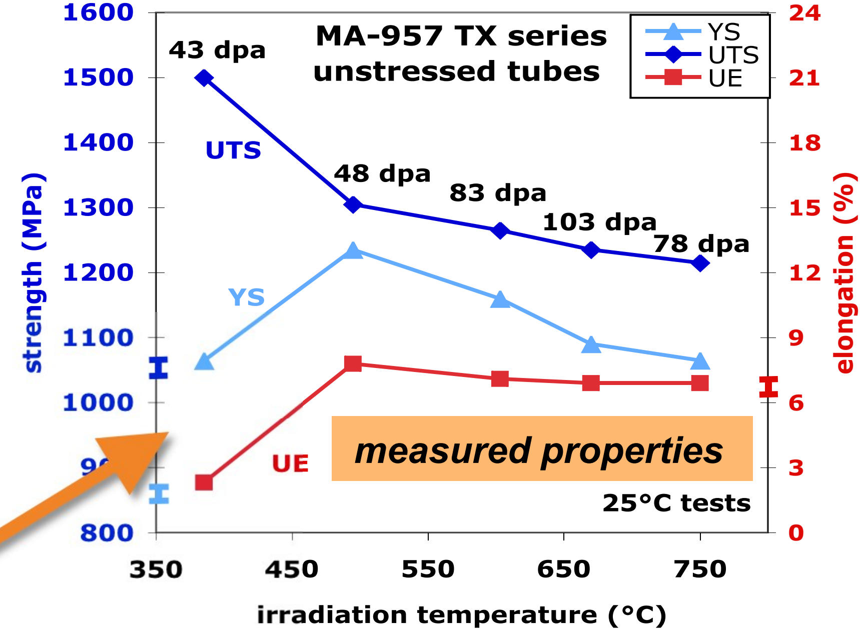
Examples of Specimens Developed During the FFTF Fast Reactor Program

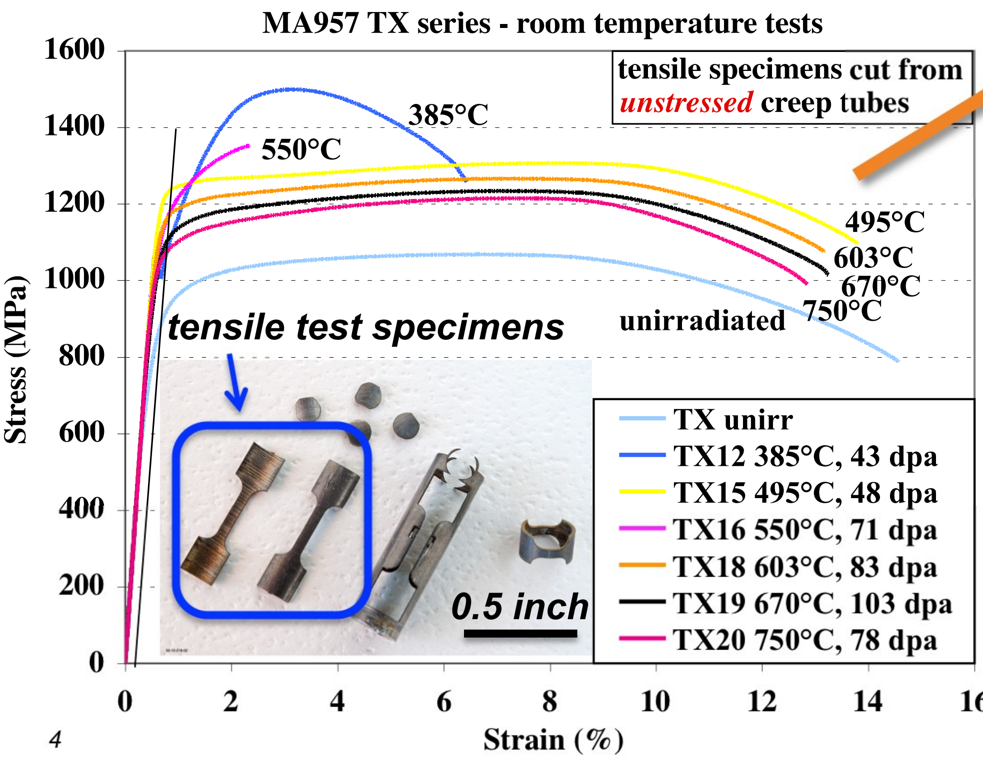
- A range of specimen geometries were developed for the fast reactor program to assess:
  - Tensile properties
  - Fracture toughness
  - Charpy response
  - In-reactor creep
  - Swelling
  - Thermal conductivity
  - Microscopy
- Some based on standard geometries while others use a unique small specimen geometry

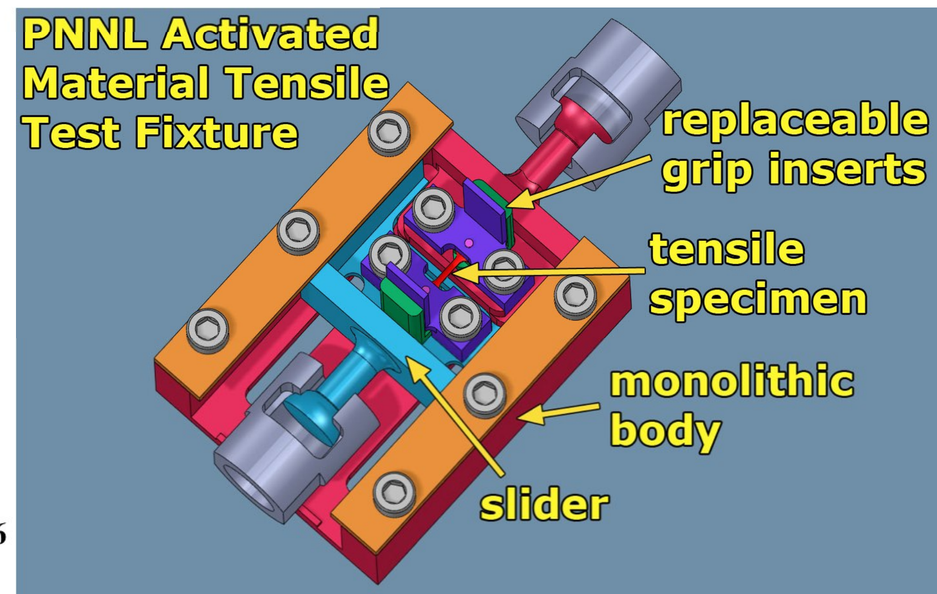


Tensile Testing of Unirradiated and Irradiated Materials

- Several studies have been performed to compare tensile properties from standard size and miniature tensile specimens.
- To obtain bulk properties, the ratio of grain size to smallest gauge dimension must be ≥10.
  - Typical stainless steel has ~25 μm grain size, so gauge thickness typically ~0.25 mm.



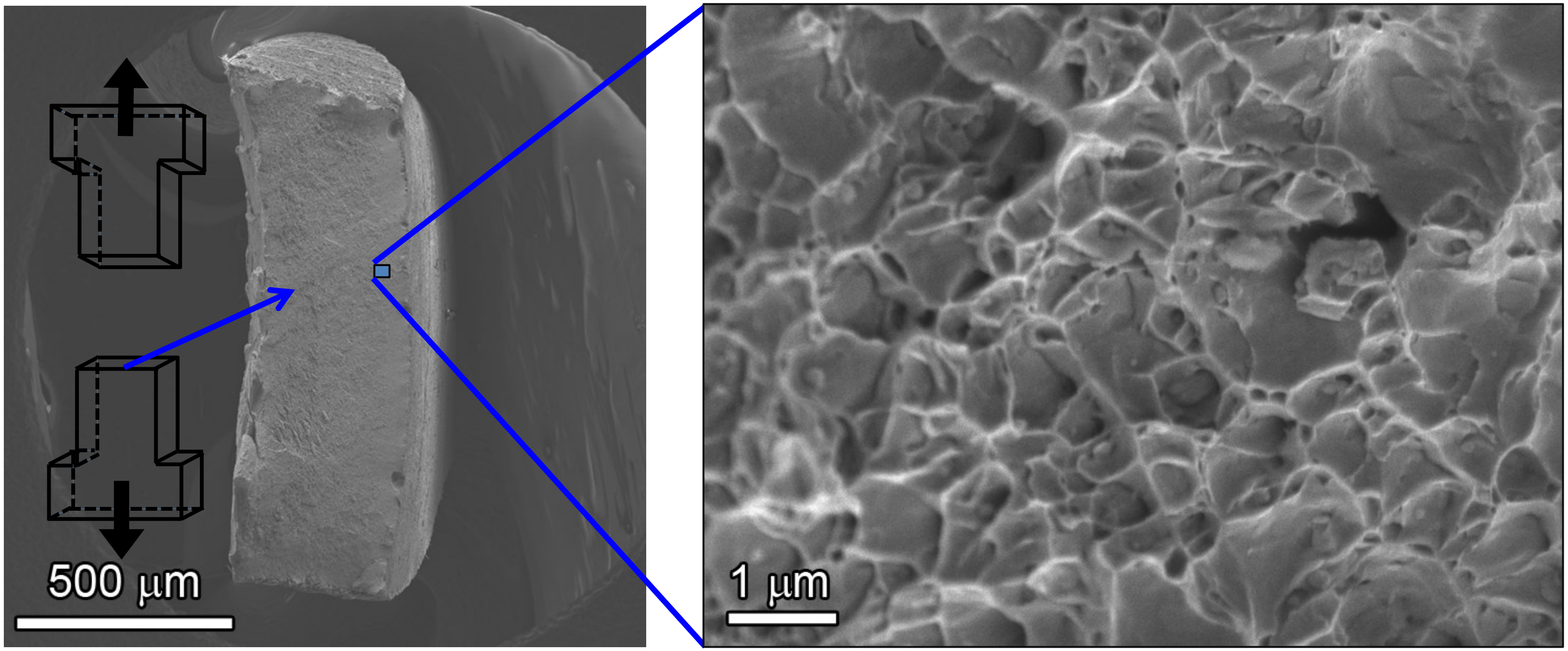




SEM Surface Examinations – Example of a Fully Ductile Failure Response

EV34 – 550°C, 113 dpa

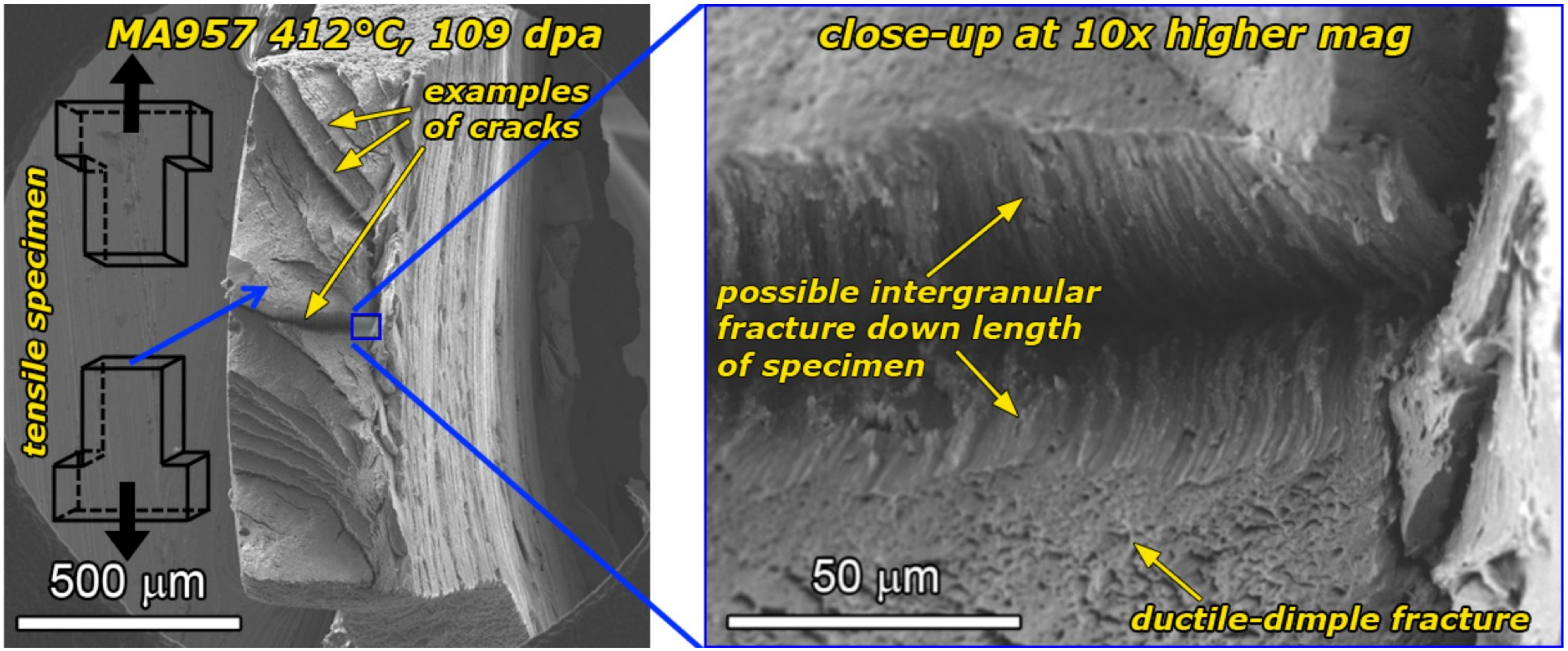
tensile specimen fracture face



After irradiation at 550°C – 750°C to 110-121 dpa, classic ductile dimple fracture was observed.

SEM Surface Examinations – Example of a Mixed Mode Failure Response

EV46 – 412°C, 109 dpa

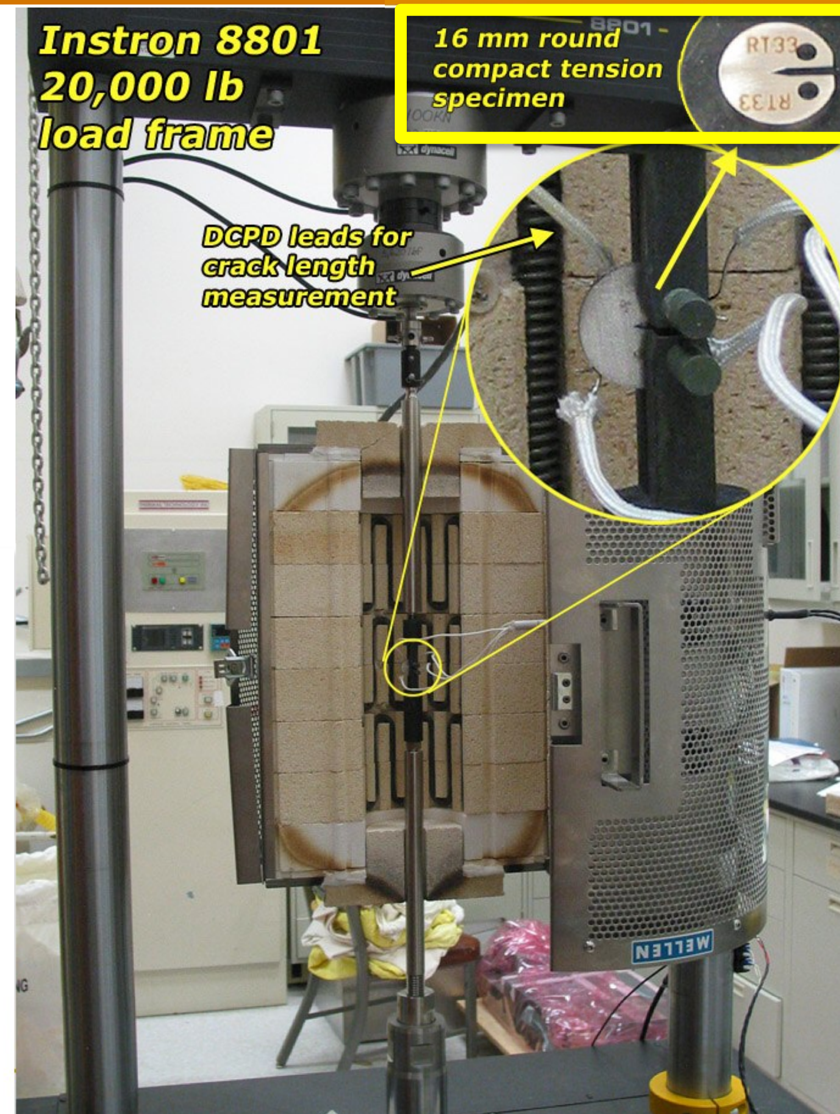


After irradiation at 412°C to 109 dpa, ductile-dimple fracture observed, but extensive splitting of the specimen along the gauge length also observed. Splitting is in the direction of the long axis of the grain boundaries. Appears to be intergranular cracking.

Fracture Toughness Testing of Unirradiated and Irradiated Materials

- J-integral fracture toughness testing allows for smaller size specimens than linear elastic fracture toughness testing, but frequently still a challenge to stay above the minimum size based on ASTM E1820.
  - Brittle materials can often be assessed using specimens ~3 mm thick x ~15 mm diameter.
  - General observation is that small specimen tests on ductile materials often produce values close to larger valid size specimens.
  - Key necessity is being able to gauge onset of brittle response as a function of dose, irradiation temperature, test temperature, etc.

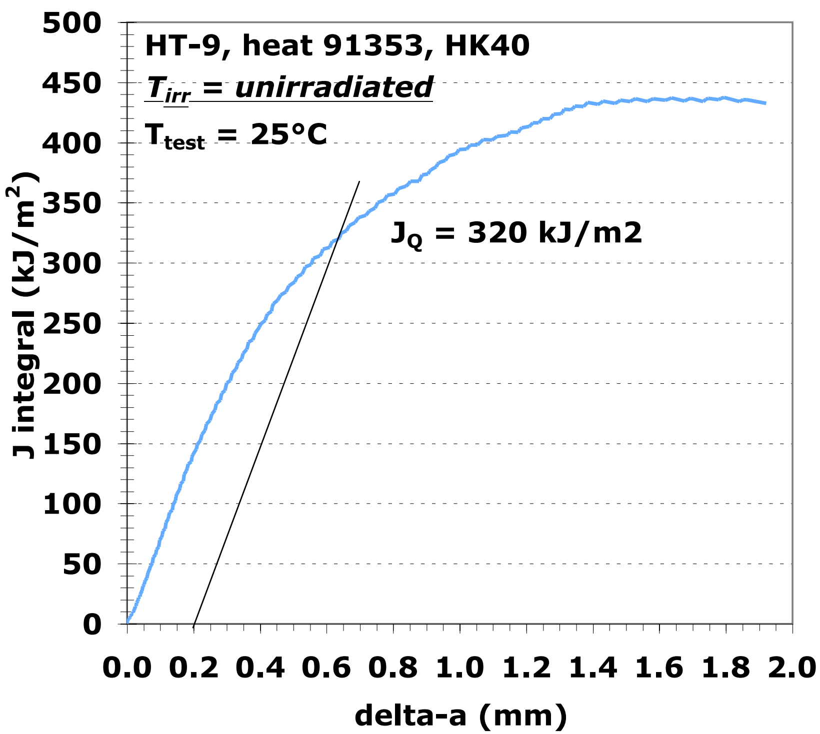
Brittle fracture of ferritic/martensitic steel fracture toughness specimen



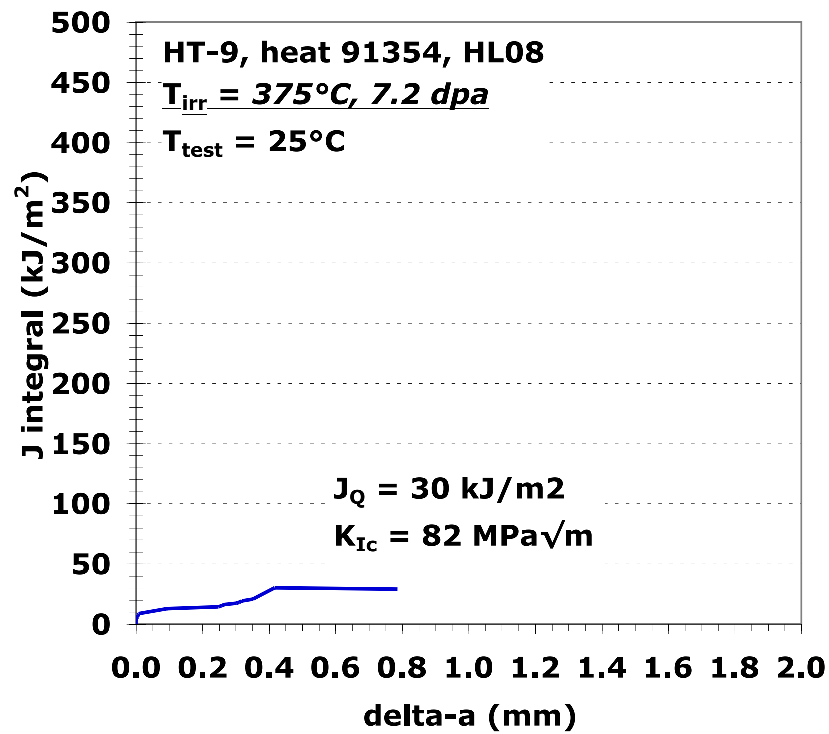
Fracture Toughness Testing of Unirradiated and Irradiated Materials

- Examples of J-integral toughness test curves of unirradiated and irradiated HT-9 tested at 25°C
  - Unirradiated HT-9 shows good plasticity, stable tearing, good toughness.
  - HT-9 irradiated at 375°C shows fully brittle response, very low toughness.

Unirradiated HT-9
stable tearing, high toughness

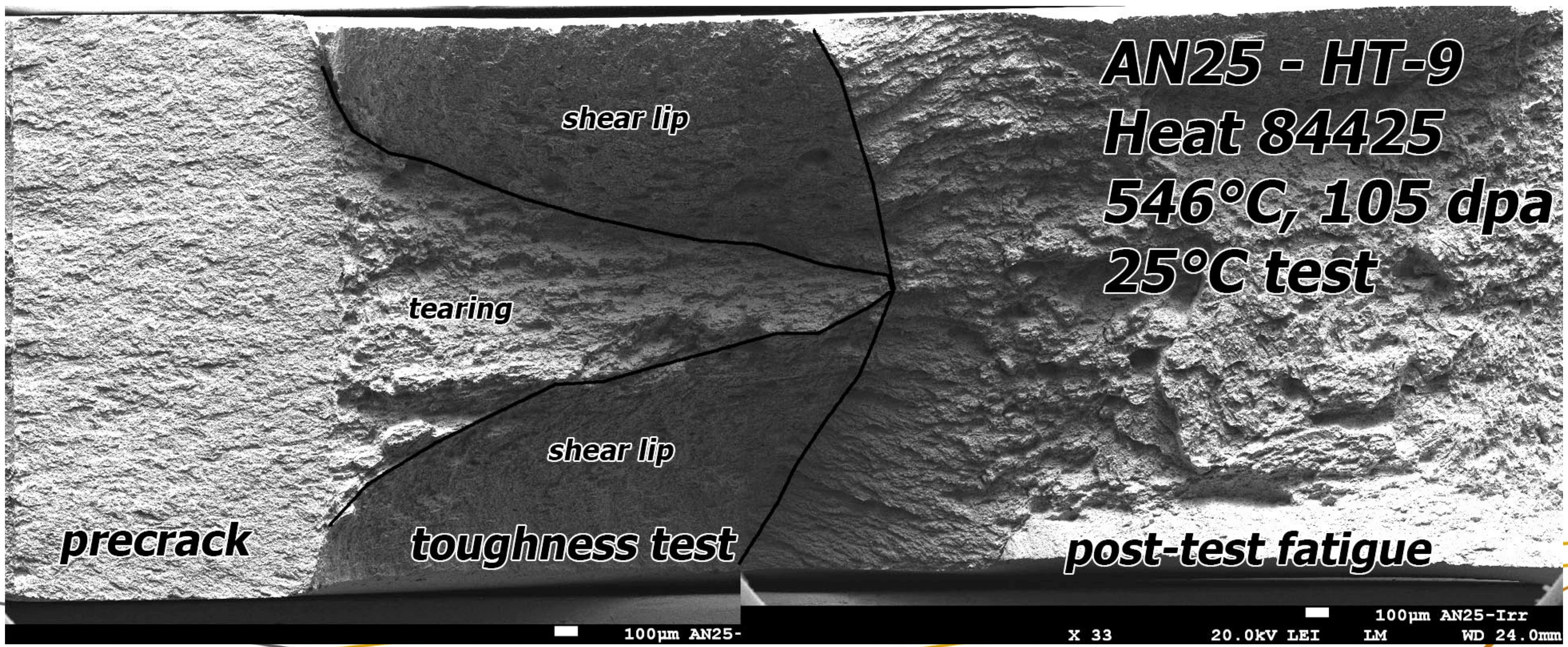


Irradiated HT-9
brittle response, low toughness



Fracture Toughness Testing – Ductile Response

- Heat 91353, T_{irr} = 546°C, 105 dpa, 25°C test, J_Q = 170 kJ/m^2 (unirradiated = ~300 kJ/m^2).
- Straight precrack, stable crack extension, shear lip formation but more tearing than found in the unirradiated material.



Fracture Toughness Testing – Brittle Response

- Heat 91354, T_{irr} = 375°C, 7.2 dpa, 25°C test, J_{KQ} = 7 kJ/m^2 (unirradiated = ~300 kJ/m^2).
- Very straight precrack, unstable crack extension, cleavage cracking

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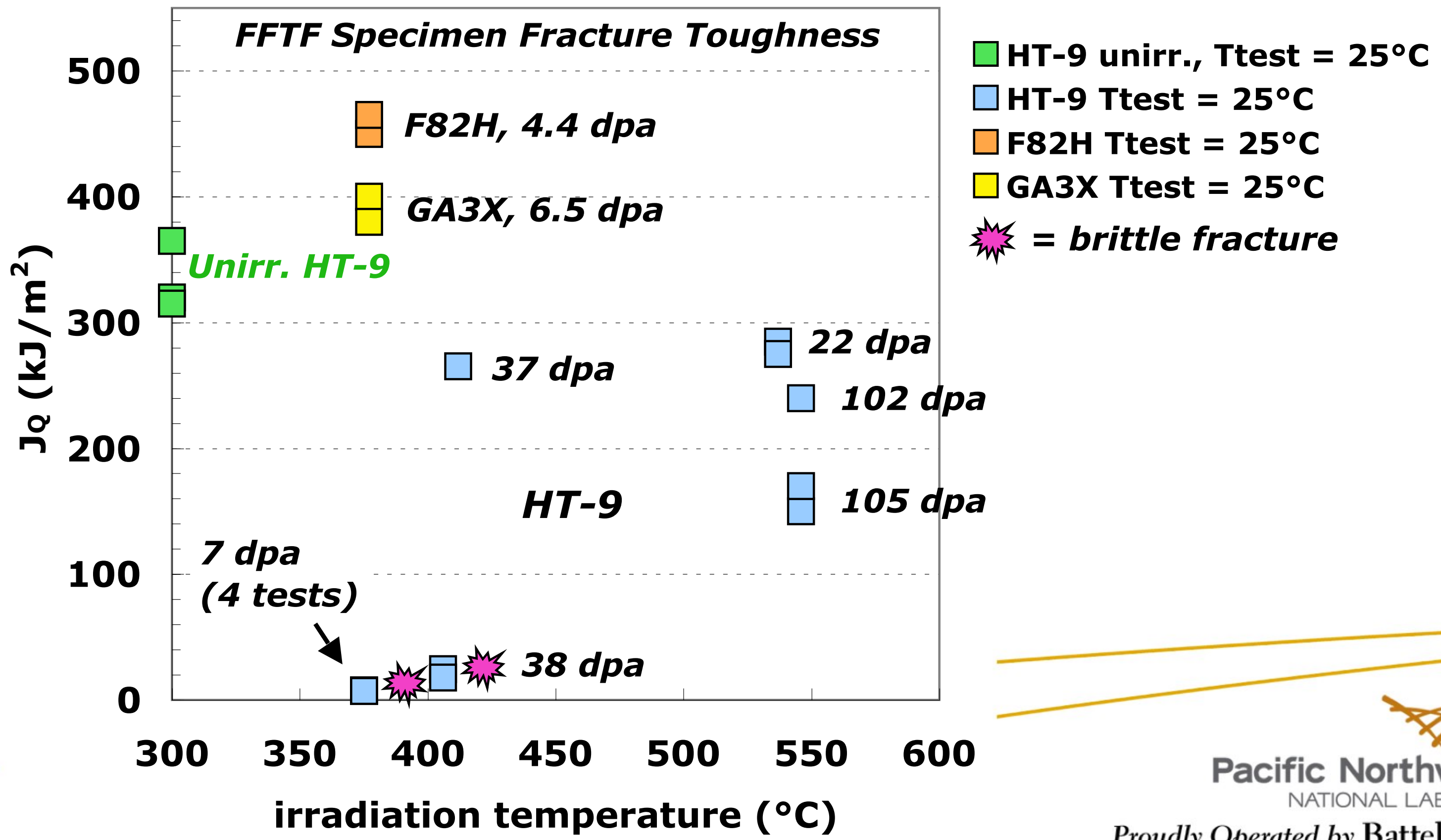
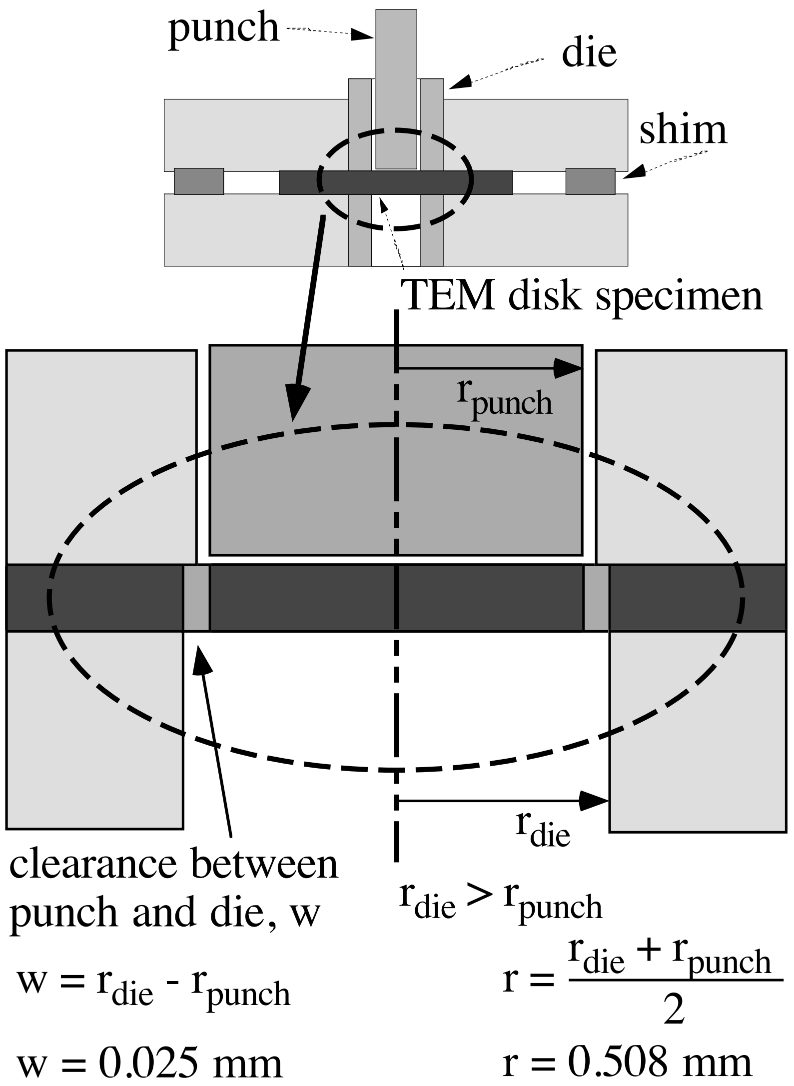
Recent Fracture Toughness Testing of HT-9, GA3X, and F82H After Irradiation at 375°C

- The 12Cr alloy HT-9 forms alpha-prime and G-phase at 375°C that locks up dislocations and forces brittle response.
- The 9Cr alloy GA3X and the 8Cr alloy F82H are relatively unaffected by irradiation form little or no alpha prime and no G-phase. Toughness remains high after irradiation.



Shear Punch Test Concept

- Estimate tensile properties from a 3 mm diameter by 0.25 mm thick disk.
- A flat face punch is driven through a specimen at a constant extension rate analogous to a tensile test.
- Produces a shear-like deformation in an annular region around the disk.
- Monitor load and punch displacement.
- Meant to serve as a screening test and not as a replacement for a tensile test.

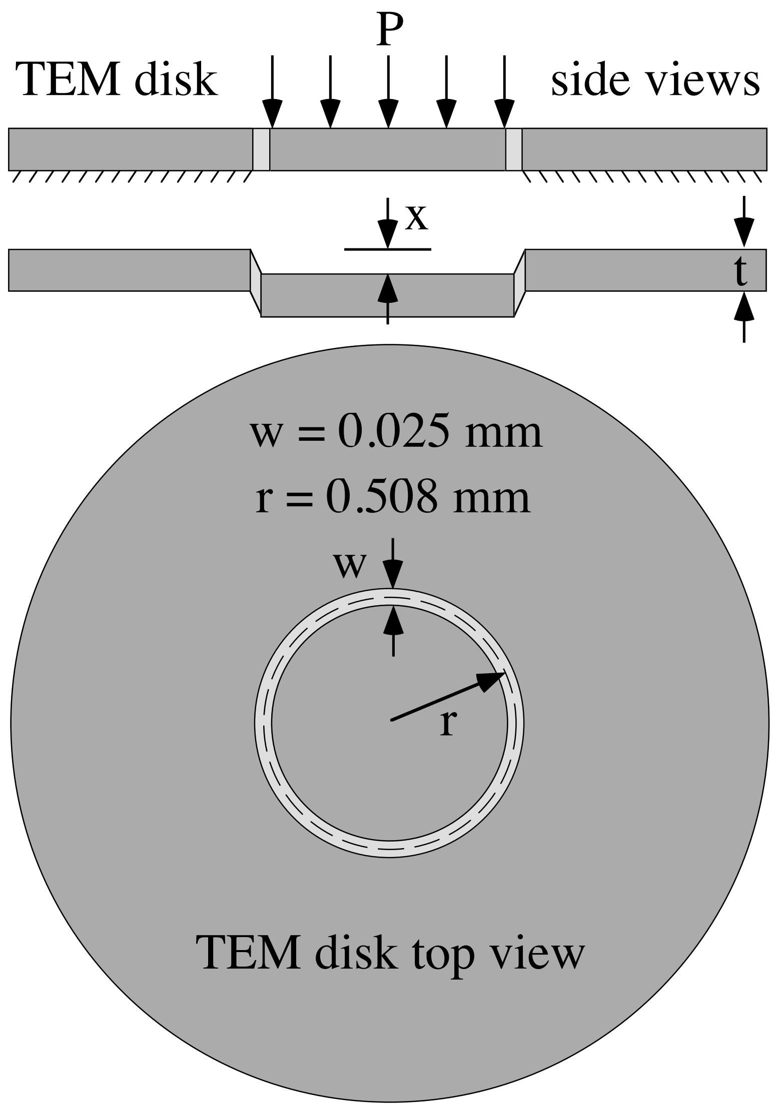


Shear Punch Test Concept

- Stress and strain approximations:

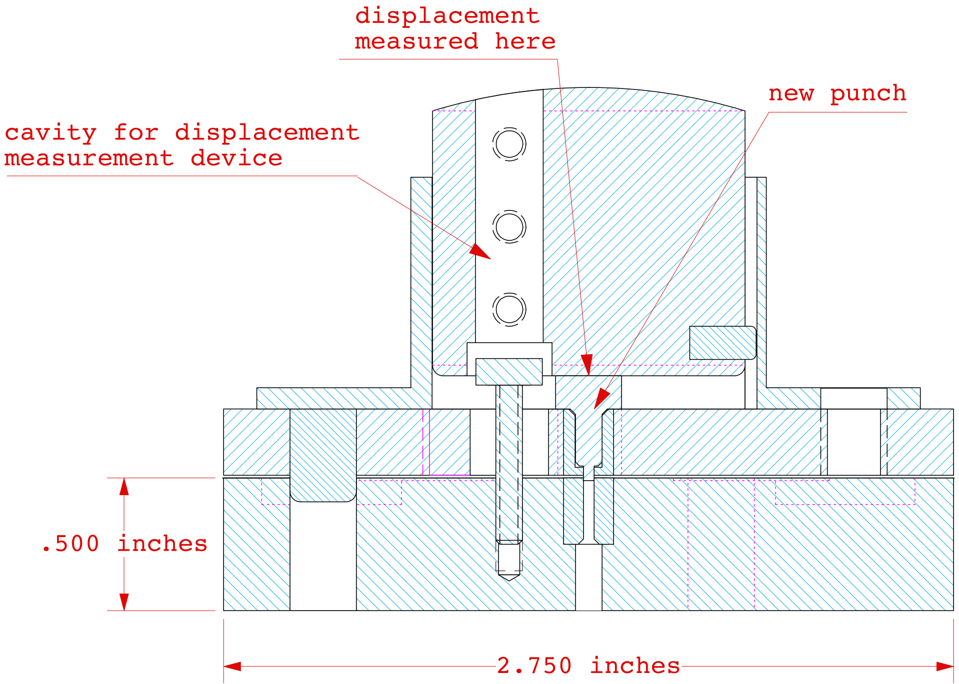
$$\tau_{y,m} = \frac{P_{y,m}}{2\pi r t} \qquad \varepsilon_{rz} = \frac{1}{2}\frac{x}{w}$$

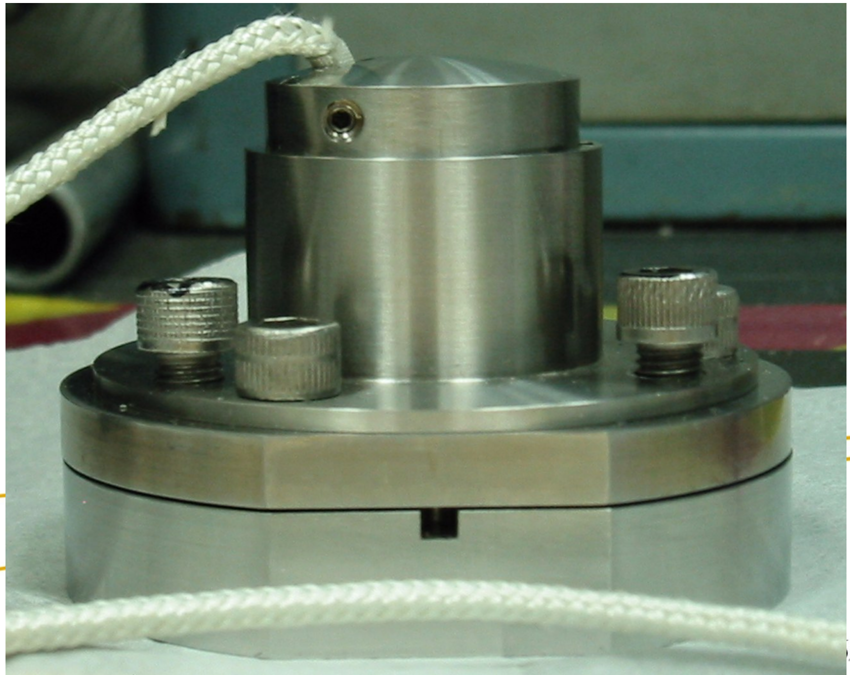
  - "P" is the load on the punch.
  - "r" is the average of the punch and die radii.
  - "t" is the specimen thickness.
  - "w" is the width of the annual deformation region.
- Initial displacement is predominantly shear, but as the test proceeds, deformation becomes more and more ligament stretching.



Shear Punch Test Concept

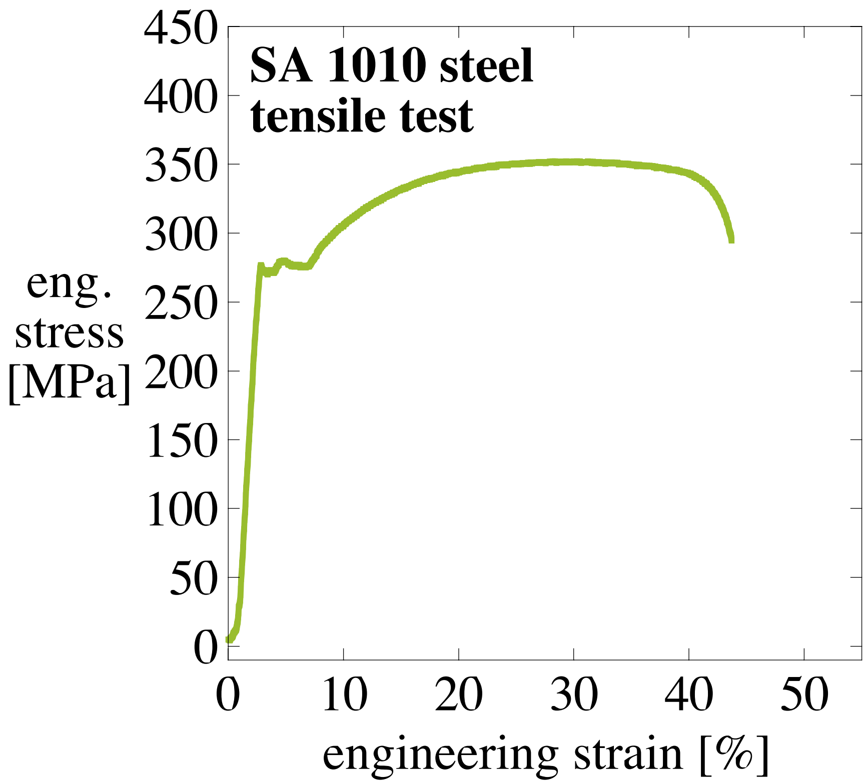
- Testing challenges
  - Accurate measurement of specimen displacement.
  - Maintaining good punch and die alignment.
  - Selection of yield strength criterion.

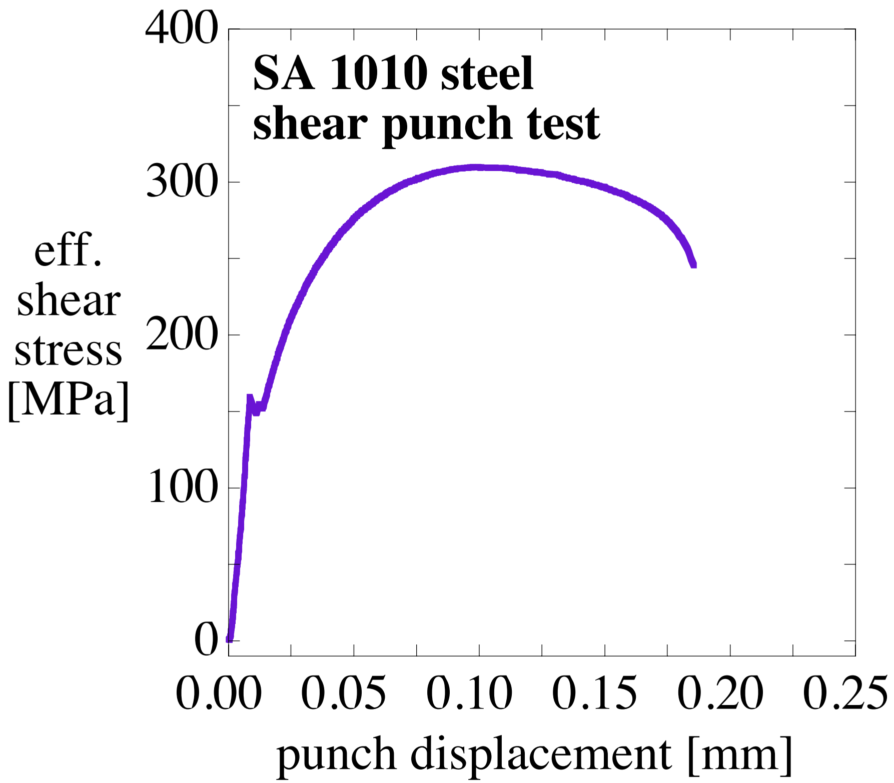




Comparative Traces: 1010 Steel (SA)

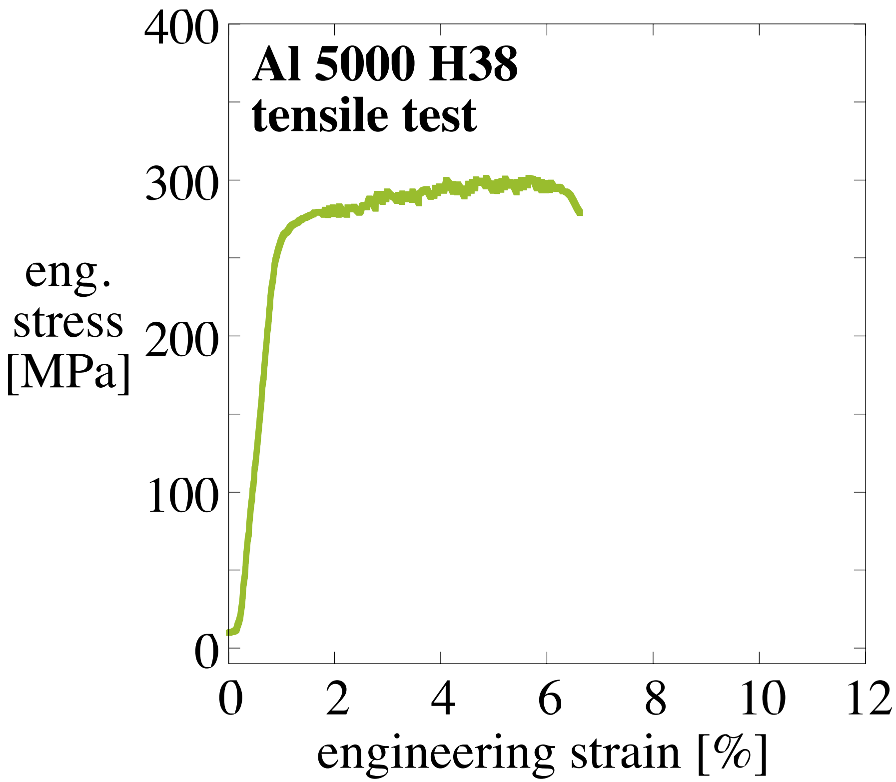
- Major features of the tensile test are reproduced in the shear punch test, in particular, the Luder's plateau is present.

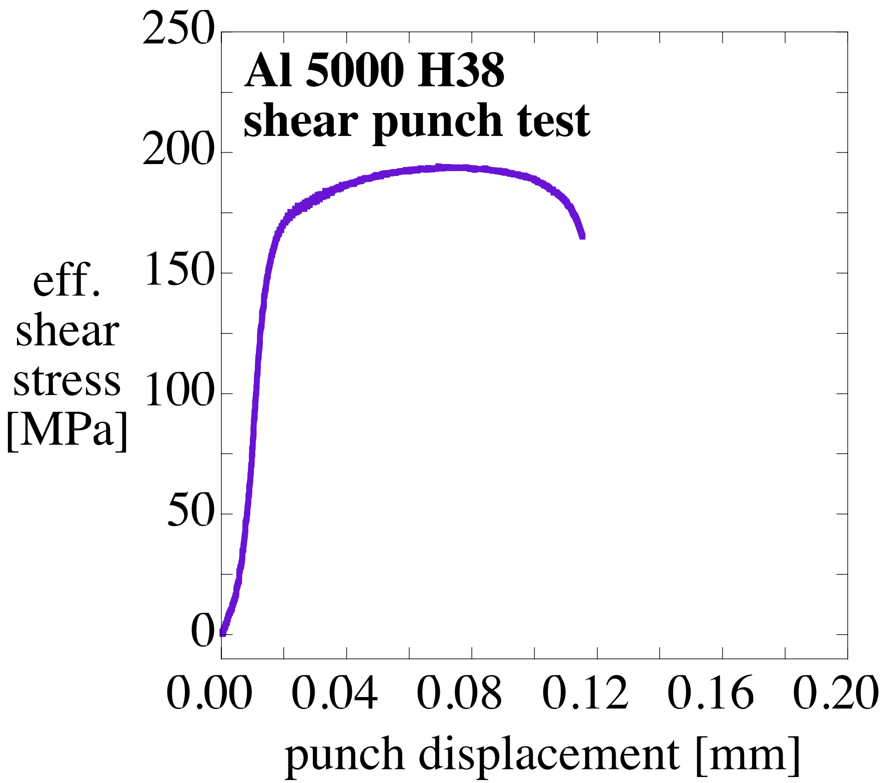




Comparative Traces: Al 5052 H38

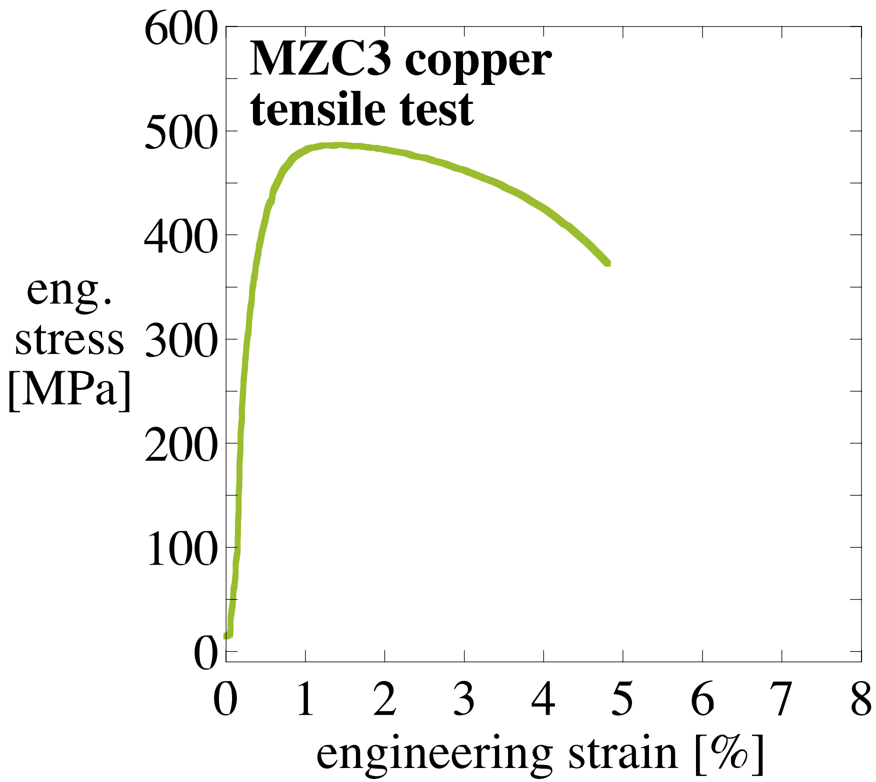
- Major features of the tensile test are reproduced in the shear punch test, in particular
  - Strain serrations are present in both traces (although very faint in the shear punch trace).
  - Similar work hardening response.

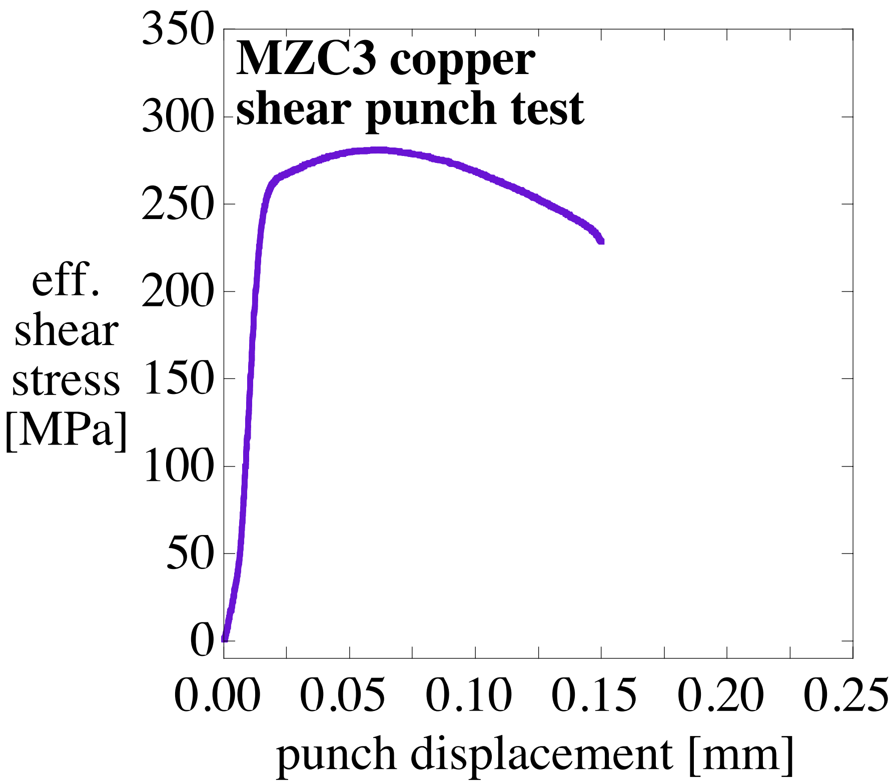




Comparative Traces: MZC3 Copper

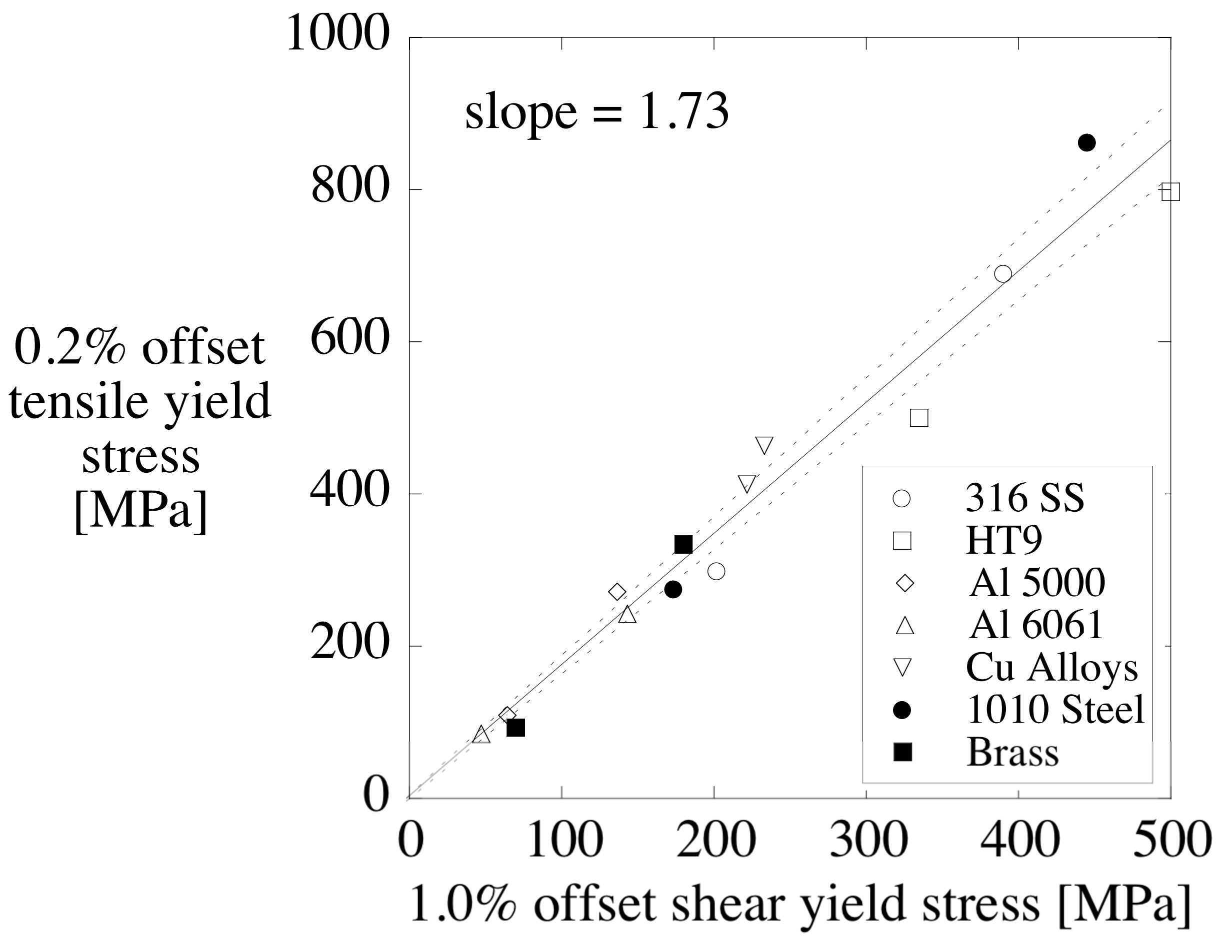
- Similar work hardening response.





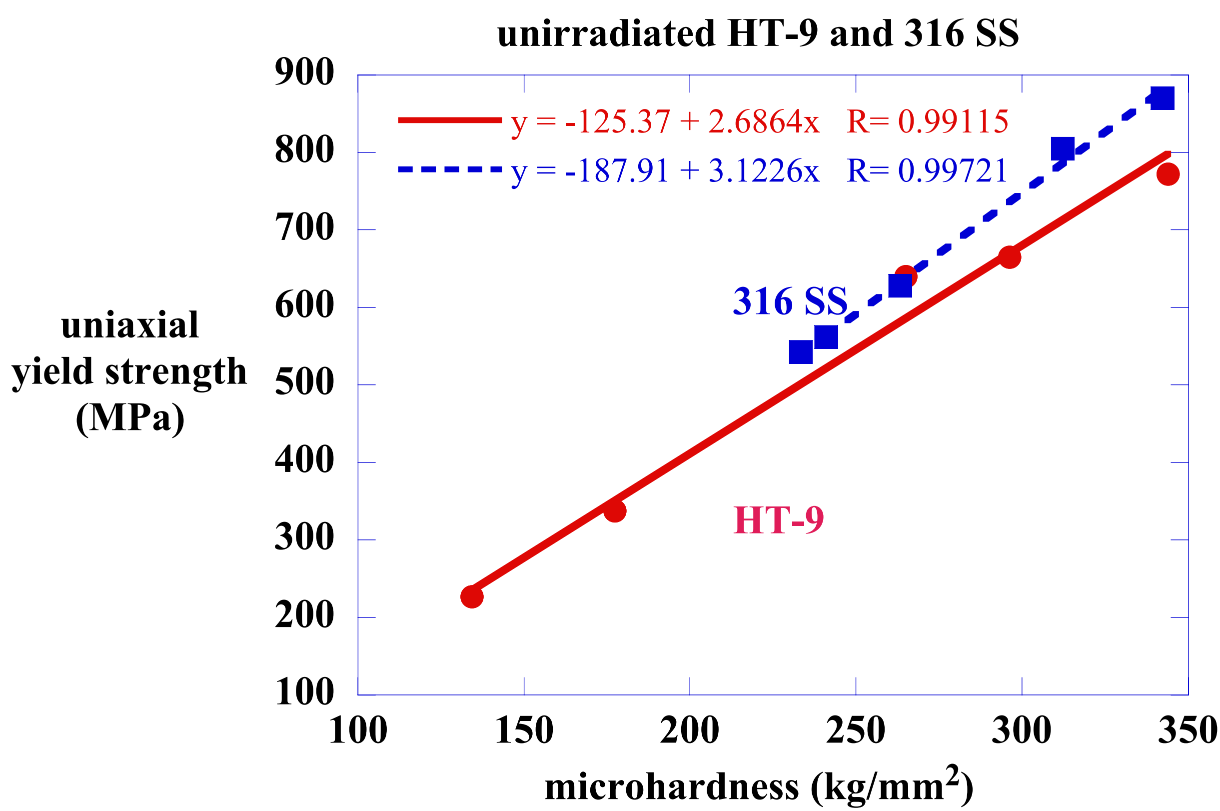
Comparison Between Tensile Yield and Shear Yield

- Shear yield obtained by measuring shear stress at 1% offset strain similar to a tensile test yield strength measurement.
- A variety of materials were tensile tested and shear punch tested at room temperature.
- Correlation slope is 1.73, matching the value predicted by the von Mises yield surface.
- Meant to serve as a screening test and not a replacement for a tensile test.



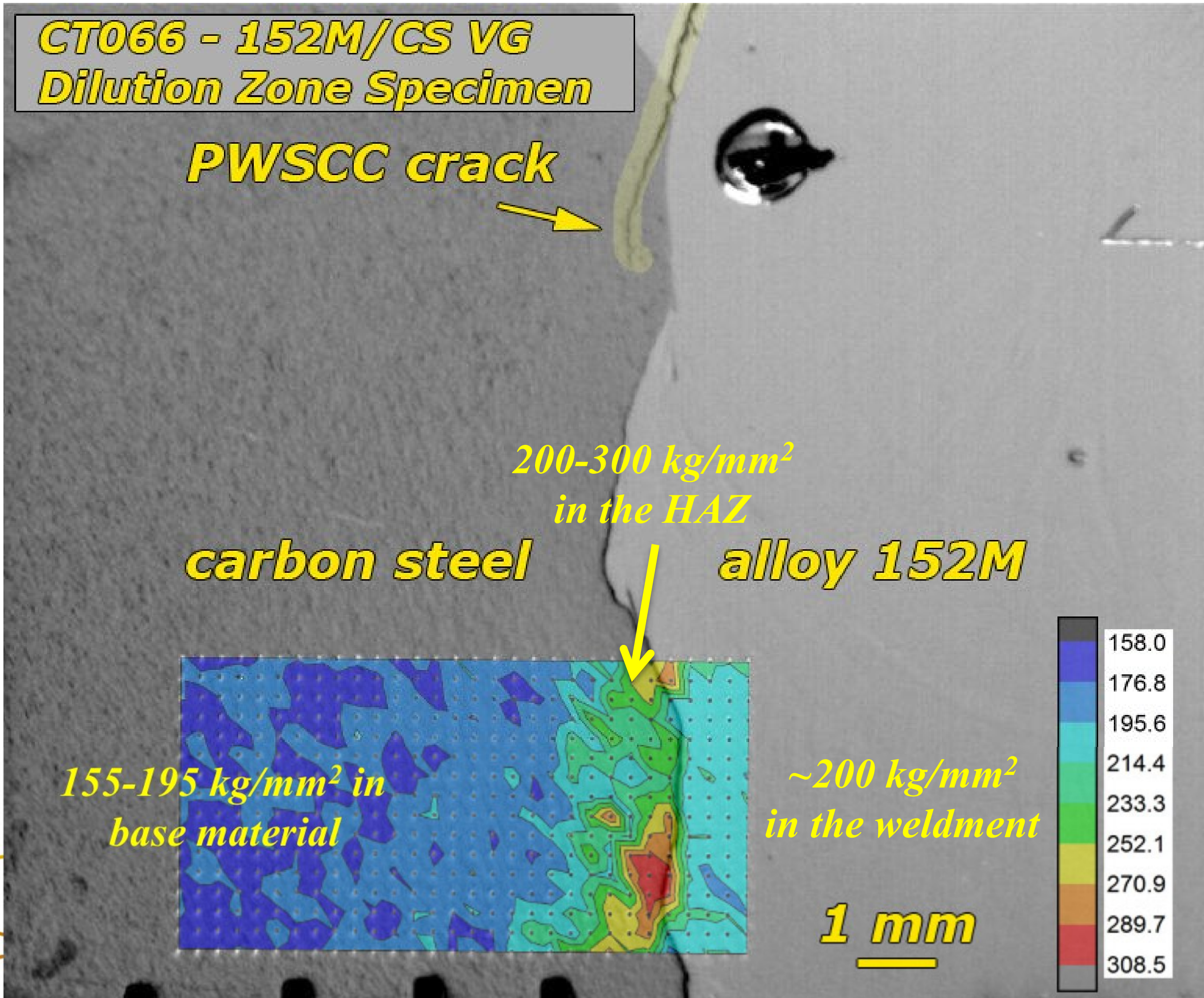
Microhardness Testing – Correlation to Tensile Properties

- It was experimentally observed in the 1950s that $3H_v$ correlated roughly with the stress measured at ~8% strain in a tensile test.
- Recent work by PNNL and others have shown that $3H_v$ also correlates roughly with the 0.2% offset yield strength.
- As with the shear punch test, yield strength vs H_v correlations can be established.
  - Most accurate when building a correlation using the material of interest.
- Provides another quick screening test for yield strength.



Microhardness Testing – Spatial Mapping

- Microhardness testing can also be used to map out spatial variation in tensile properties.
- In this example, microhardness has been used to identify an increase in hardness in the heat affected zone (HAZ) in a dissimilar metal interface.
- This HAZ was found to be susceptible to stress corrosion cracking.



Summary

- Miniature specimen testing is a mature field in the area of radiation effects research.
- A wide variety of miniaturized tests have been developed and used successfully for probing the effects of irradiation on material properties.
- Two basic classes of small specimen tests exist:
  - Miniature size versions of standard test geometries that directly produce engineering relevant properties. Specimen size must be within certain limits to produce bulk properties.
  - Non-standard geometries where engineering relevant properties are estimated. Often based on very small size specimens. These often serve as screening tests.