Effect of H-charging on dislocation multiplication in pre-strained super duplex stainless steel

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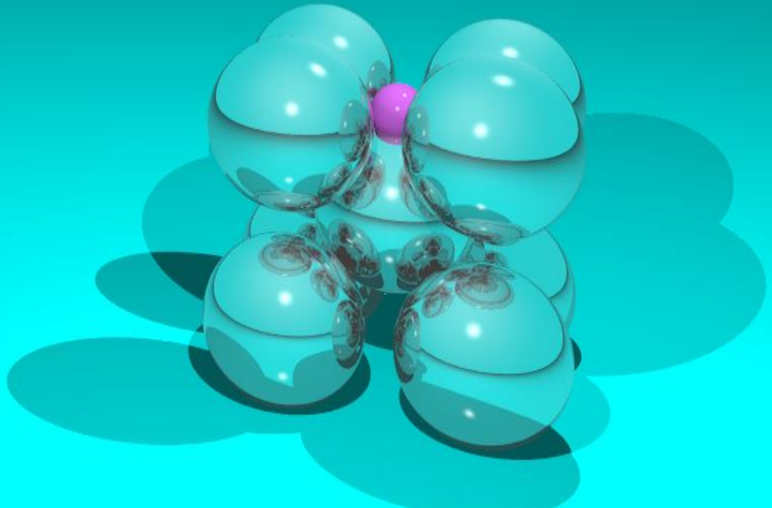

Postgraduate Research Symposium on Ferrous Metallurgy, Feb 2018

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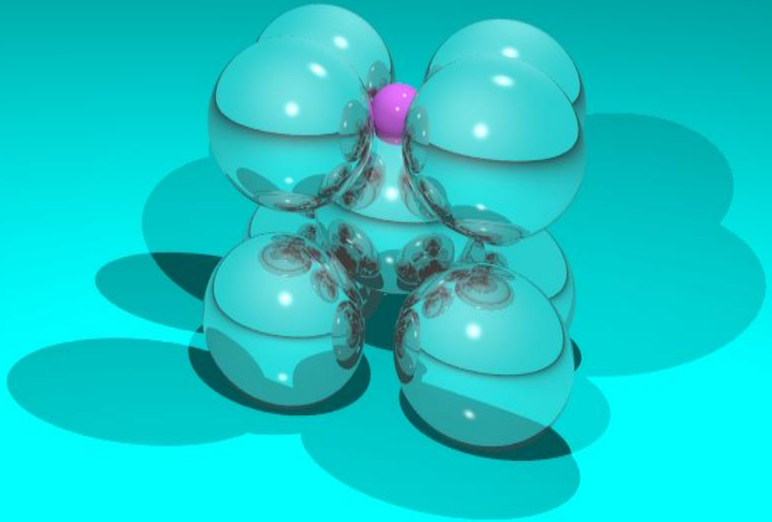
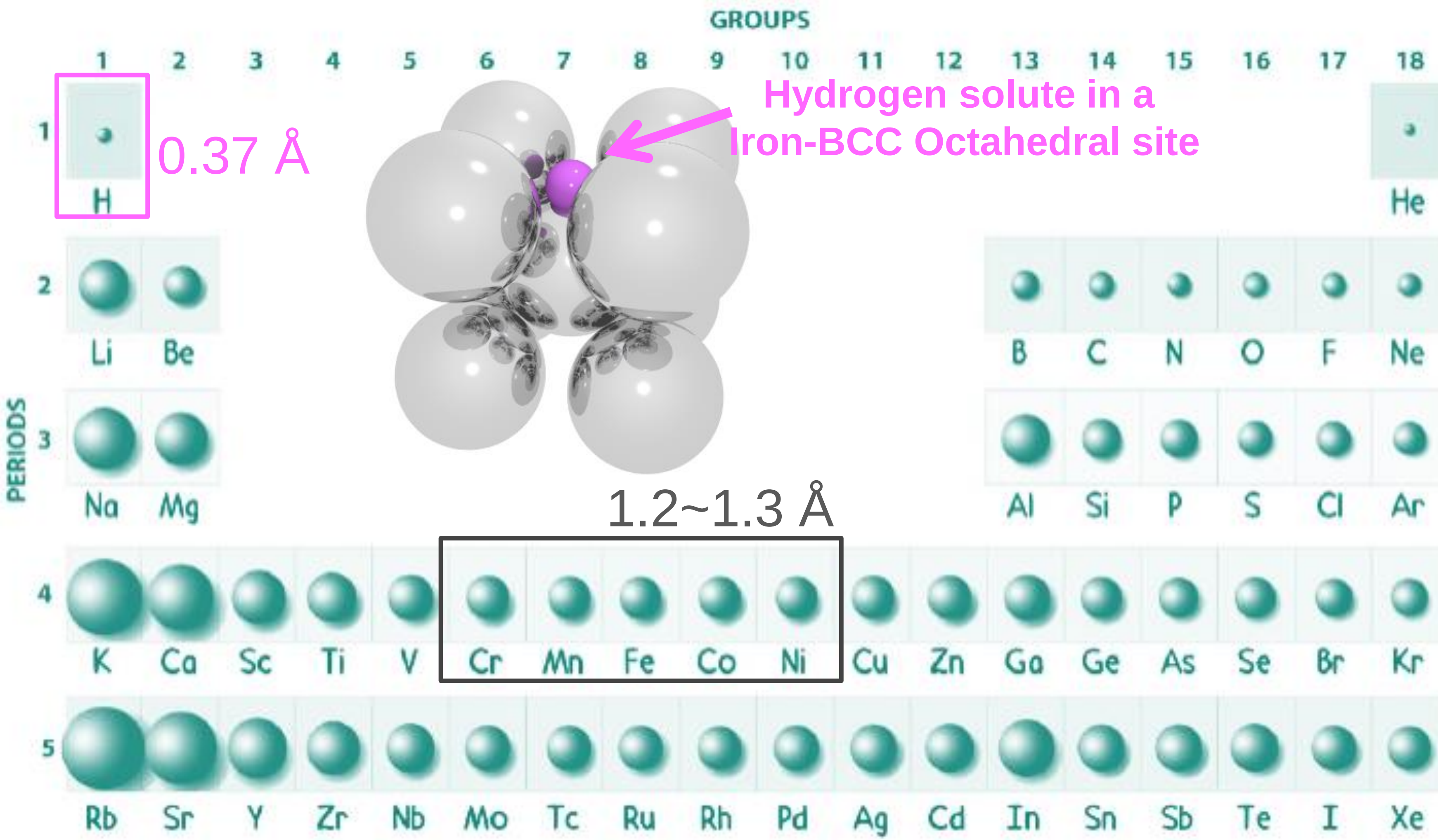
Background



Physical feature of hydrogen atom



Periodic table adopted from: *http://slideplayer.com/slide/7280859/*

Hydrogen in metals

Hydrogen may be introduced during:

- Casting
- Welding

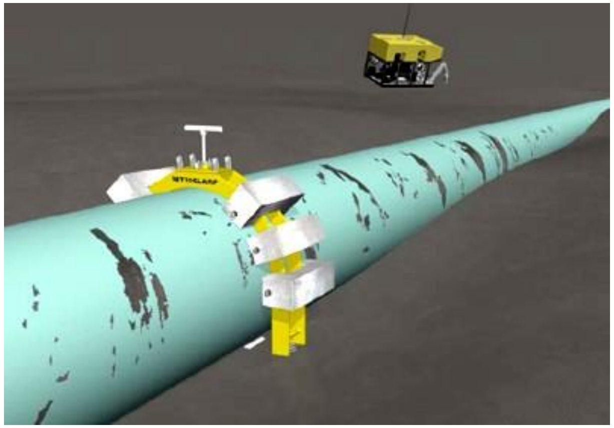
Processing

- Cathodic protection

Servicing

Continuously introducing hydrogen into materials

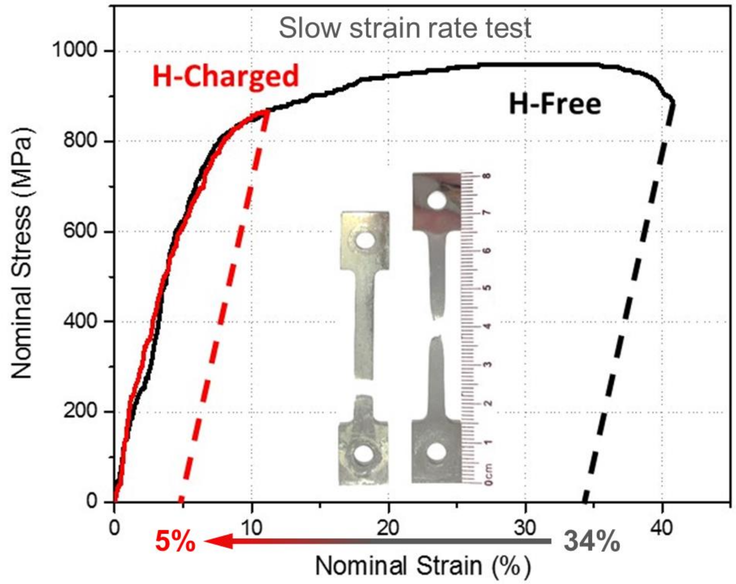
casting

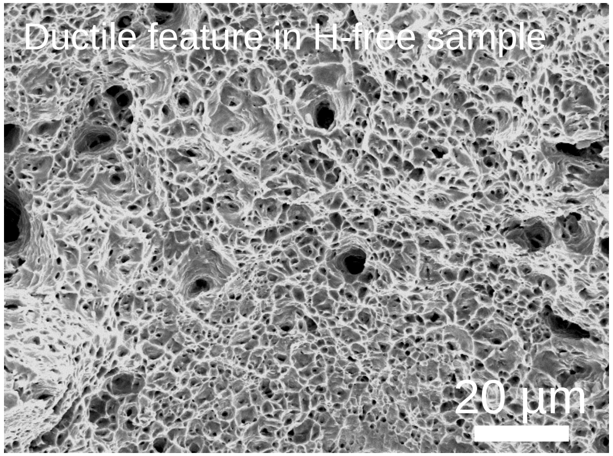


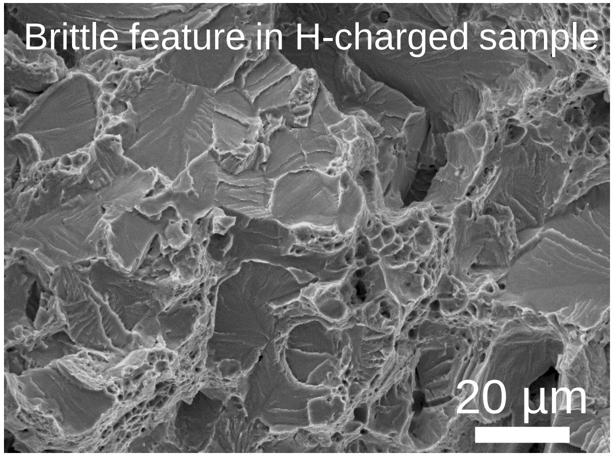
cathodic protection

welding

Effect of hydrogen charging on ductility







Materials: Zeron 100; sample gauge volume: 35×5×1.2 mm^3; hydrogen charge parameter: two weeks charging in 3.5 wt. % NaCl at 1.2 V with 50°C heating; strain rate during tensioning: 10^{-4} s^{-1}

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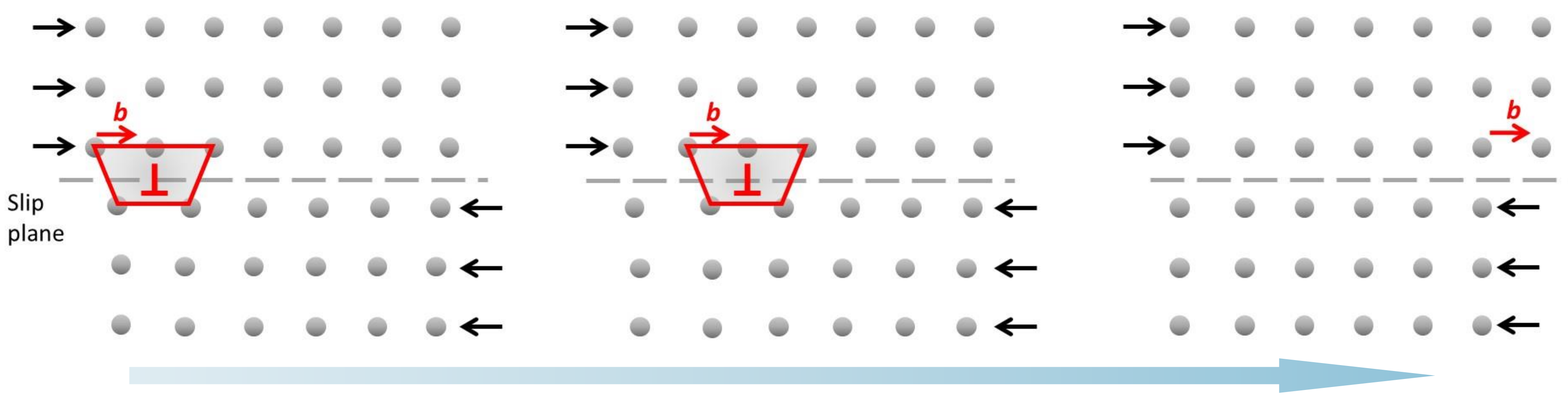
Defects in metals

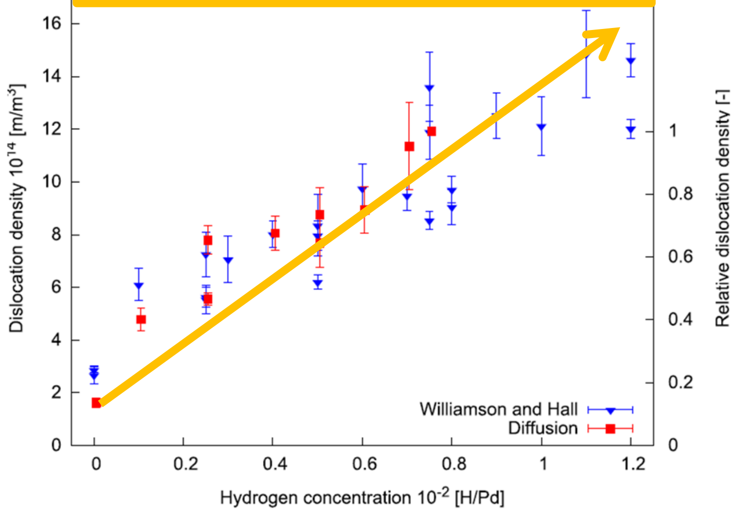
- Point defect (e.g. vacancy)
- Linear defect – dislocations
- Interfacial defect – boundaries or stacking faults



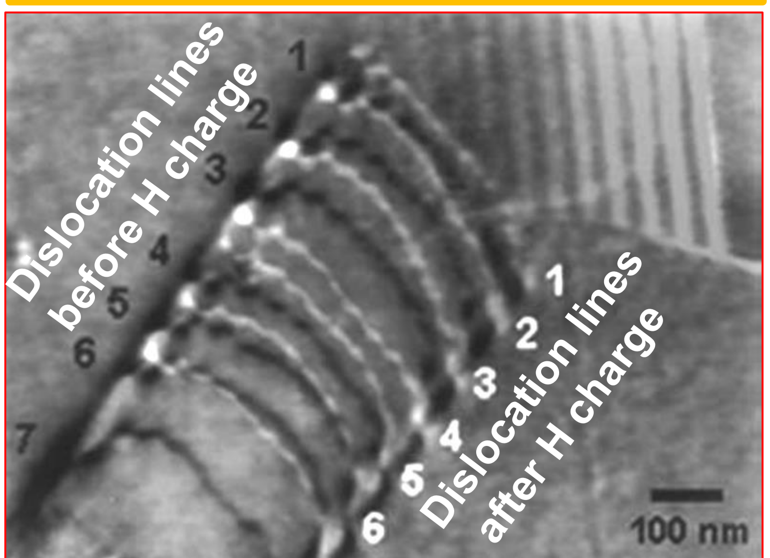
Dislocation movement picture adopted from: *https://www.nde-ed.org/EducationResources/CommunityCollege/Materials/Graphics/DislocationMovement.gif*

H-dislocation interaction

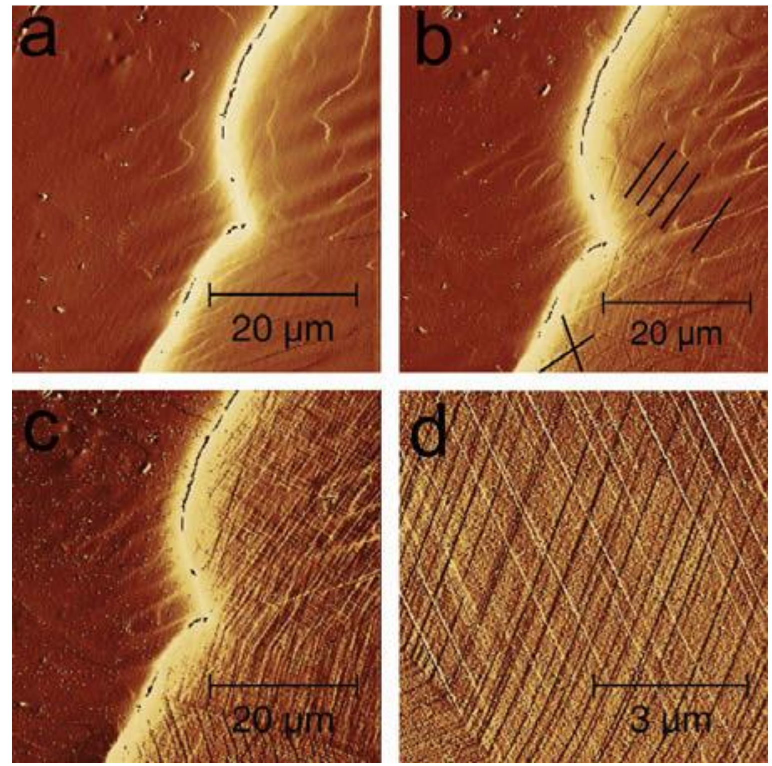
1. Hydrogen increases dislocation density in cold-rolled Pd – x-ray diffraction



2. Hydrogen activates dislocation movement - TEM



3. Hydrogen activates slip line formation in super duplex stainless steel - AFM



Ref:
1. Deutges et al. 2015, Acta Mater.
2. Ferreira et al. 1998, Acta Meter.
3. Barnoush et al. 2010, Scr. Mater.

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Advantages of Neutron Diffraction

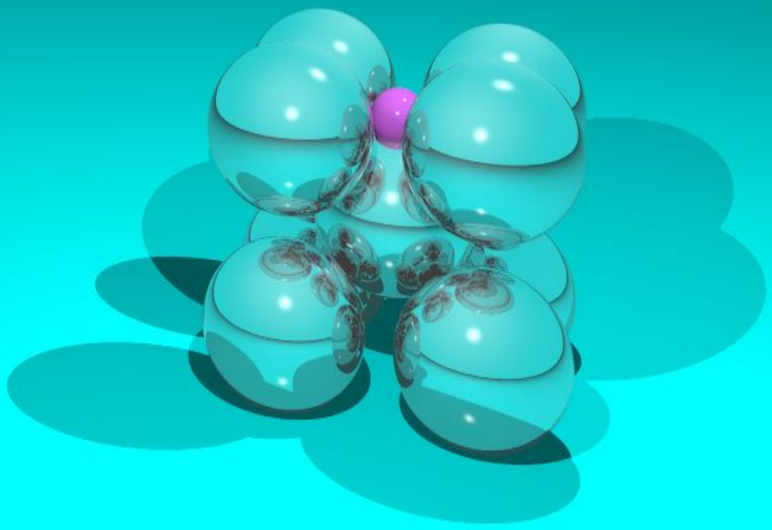
Challenges

- Limited to (near) surface observation
  - e.g. X-ray diffraction, atomic force microscopy, TEM
- Long scanning time is required due to low beam flux, which may lead to measurement errors from surface hydrogen escape
- Require high standard for sample preparation

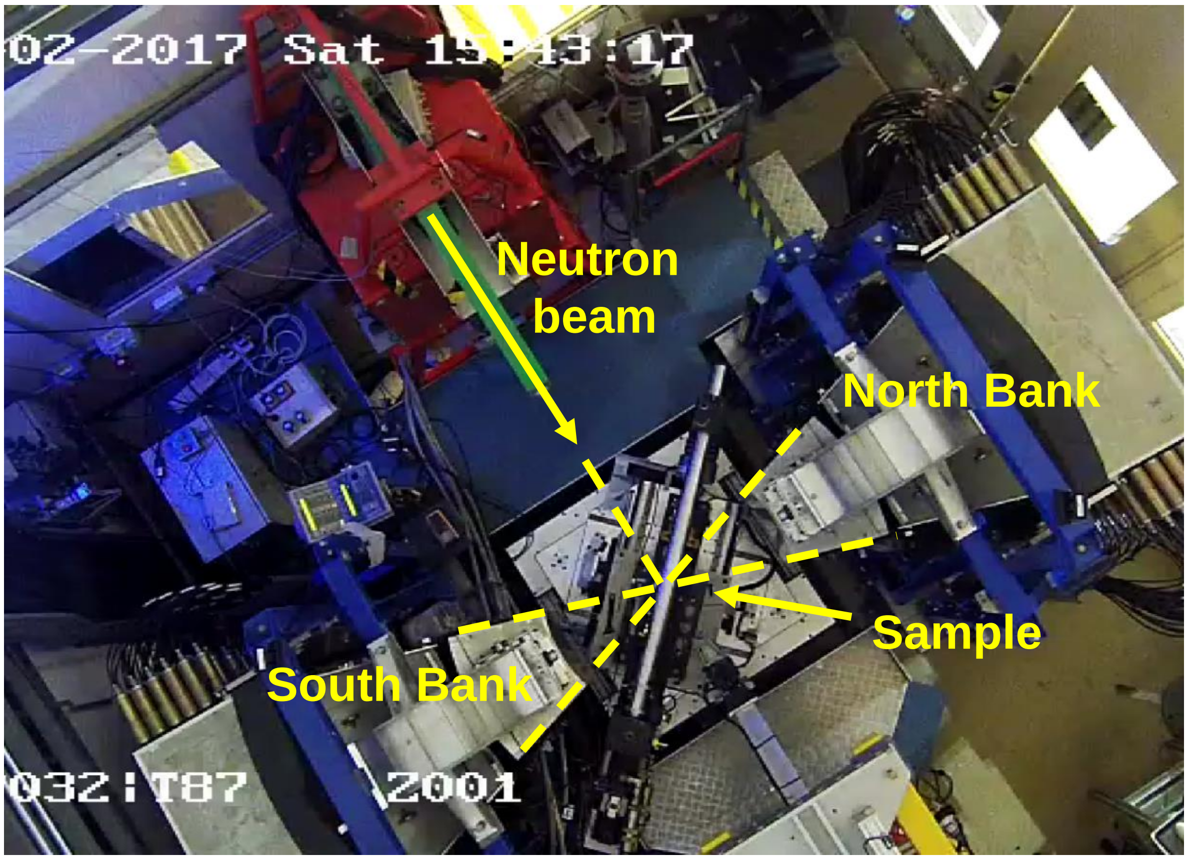
Neutron diffraction

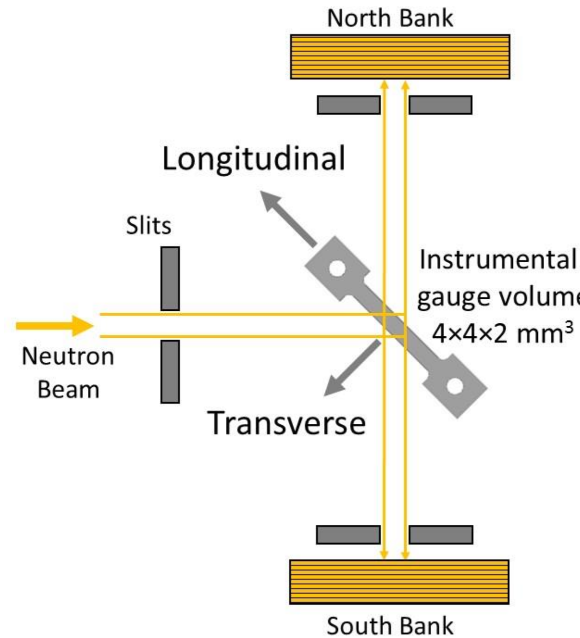
- **Bulk information** can be obtained as neutron has high penetration capability
- Reduced scanning time owning to high beam flux in Engin-X, which **minimises the hydrogen escape** during scanning
- Relatively **simple routine for sample preparation**

Methodology



Neutron diffraction setup





ENGINE-X, ISIS, UK

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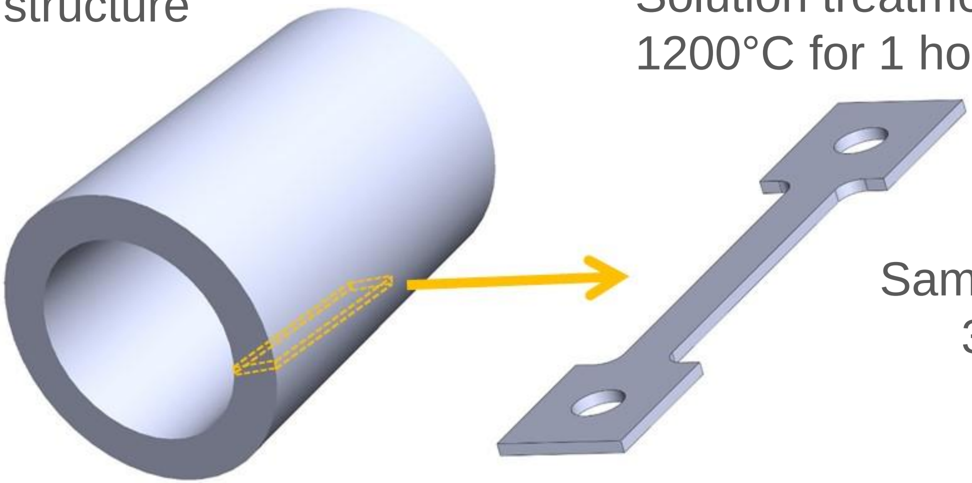
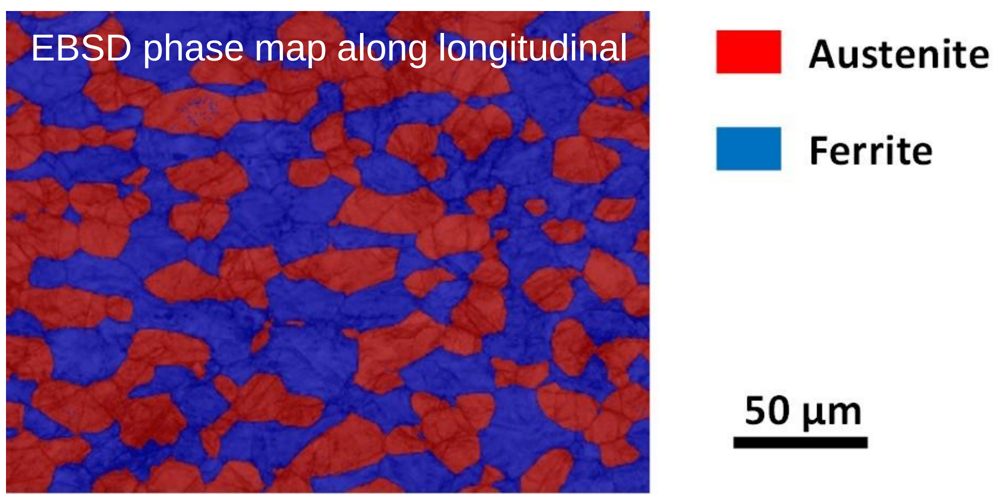
Sample preparation

Pipe structure

Solution treatment
1200°C for 1 hour

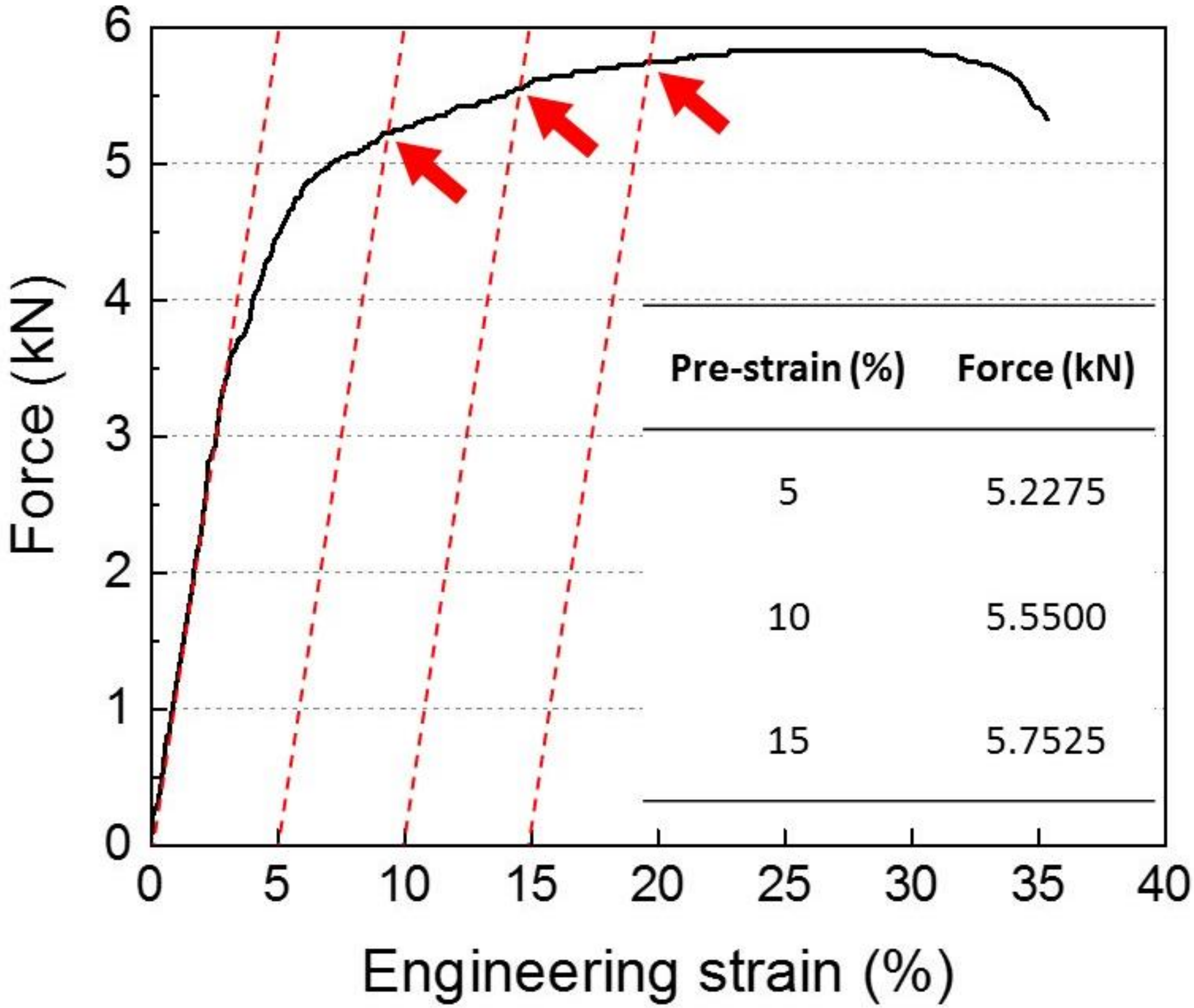


Sample gauge volume
35×5×1.2 mm^3



Step size: 0.3 µm; Acc. voltage: 20 kV

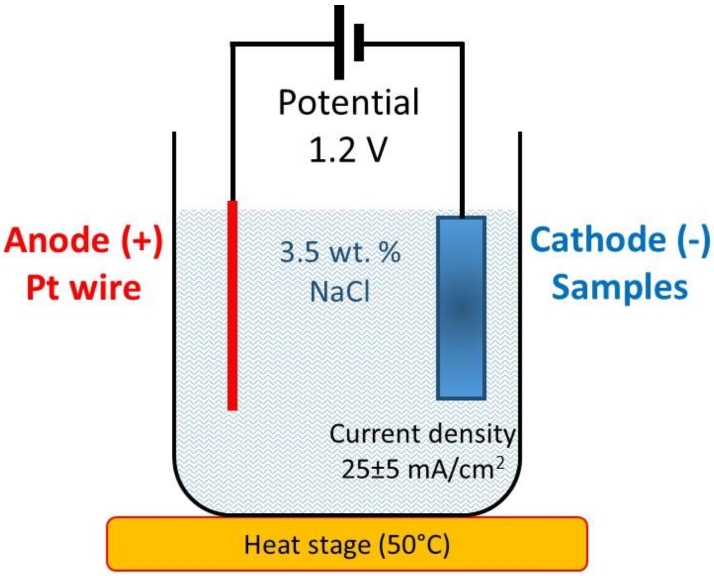
Preparation of pre-strain samples

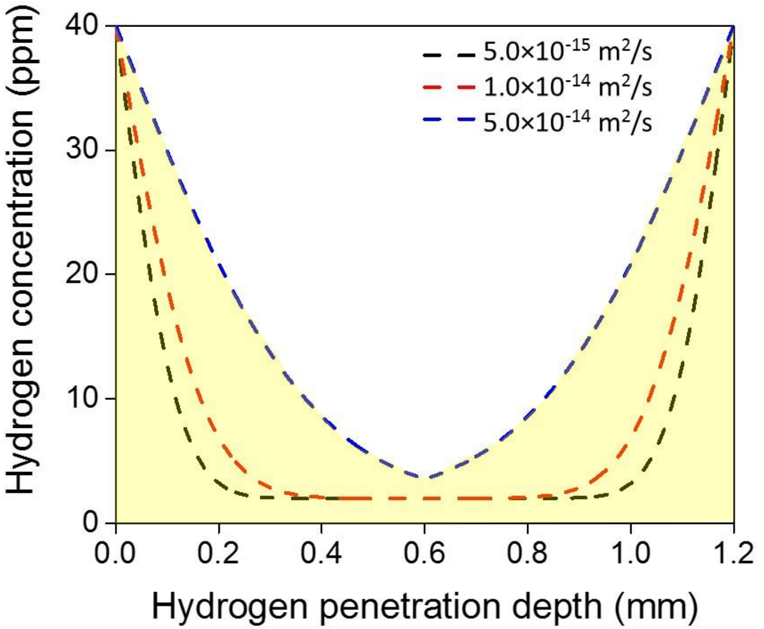


Pre-strain (%)	Force (kN)
5	5.2275
10	5.5500
15	5.7525

Tensile curve of sample engineering strain (%) against force (kN); the pre-strain of 5%, 10% and 15% can be achieved with 5.2275, 5.5500 and 5.7525 kN loads; uncertainty of pre-strain is measured as 0.3%.

Hydrogen charging cell





H-charging parameter:

- 3.5 wt. % NaCl solution
- 50° C heating environment
- 10 days H-charging
- 25±5 mA/cm² current density.

Measurement of dislocation density

Williamson-Hall equation

$$\frac{\Delta d}{d} = \frac{d}{t} + \varepsilon \quad (1)$$

This term can be ignored due to its small contribution

Faulkner equation (total elastic energy)

$$U = \frac{15}{4}\frac{E}{(1+v)}\varepsilon^2 = \frac{15}{4}\frac{E}{(1+v)}\left(\frac{\Delta d}{d}\right)^2 \quad (2)$$

The elastic energy per unit length of dislocation (u)

$$u = \frac{Gb^2}{4\pi} ln\frac{r_1}{b} \quad (3)$$

Together (1-3), increased **dislocation density can be derived**

$$\boldsymbol{\rho_b = \frac{U}{u} = \frac{15E}{2Gb^2(1+v)}\left(\frac{\Delta d}{d}\right)^2} \quad (4)$$

Peak broadening term

where d is interplanar spacing; t is grain size; ε is elastic strain; E is the Young's modulus; v is Poisson's ratio; G is shear modulus; r_1 is the effective elastic field radius at a dislocation core; and b is the burger's vector. Assumptions are made that r_1=100 nm, b=0.248 nm for ferrite and b=0.254 nm for austenite; The value $ln\frac{r_1}{b} \approx 2\pi$.

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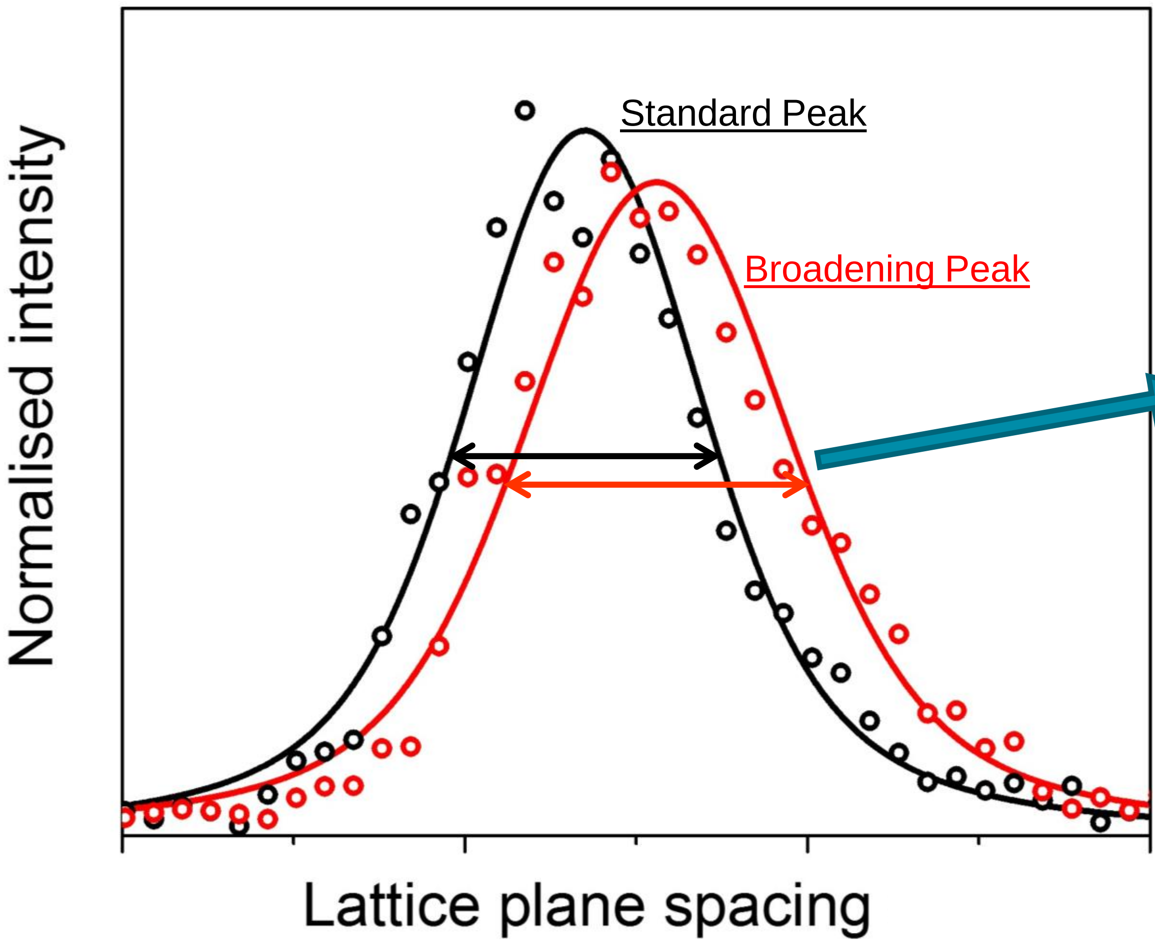
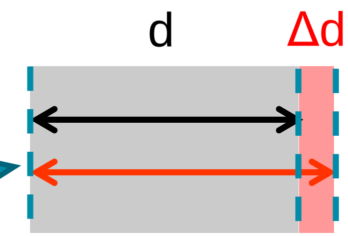
Measurement of dislocation density



FWHM of Standard peak

Represents a 10^{12} m^{-2} dislocation density

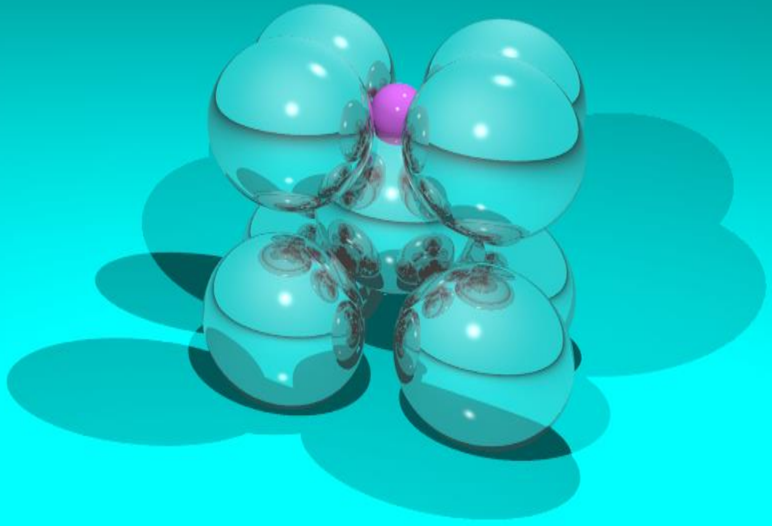


FWHM of Broadening peak

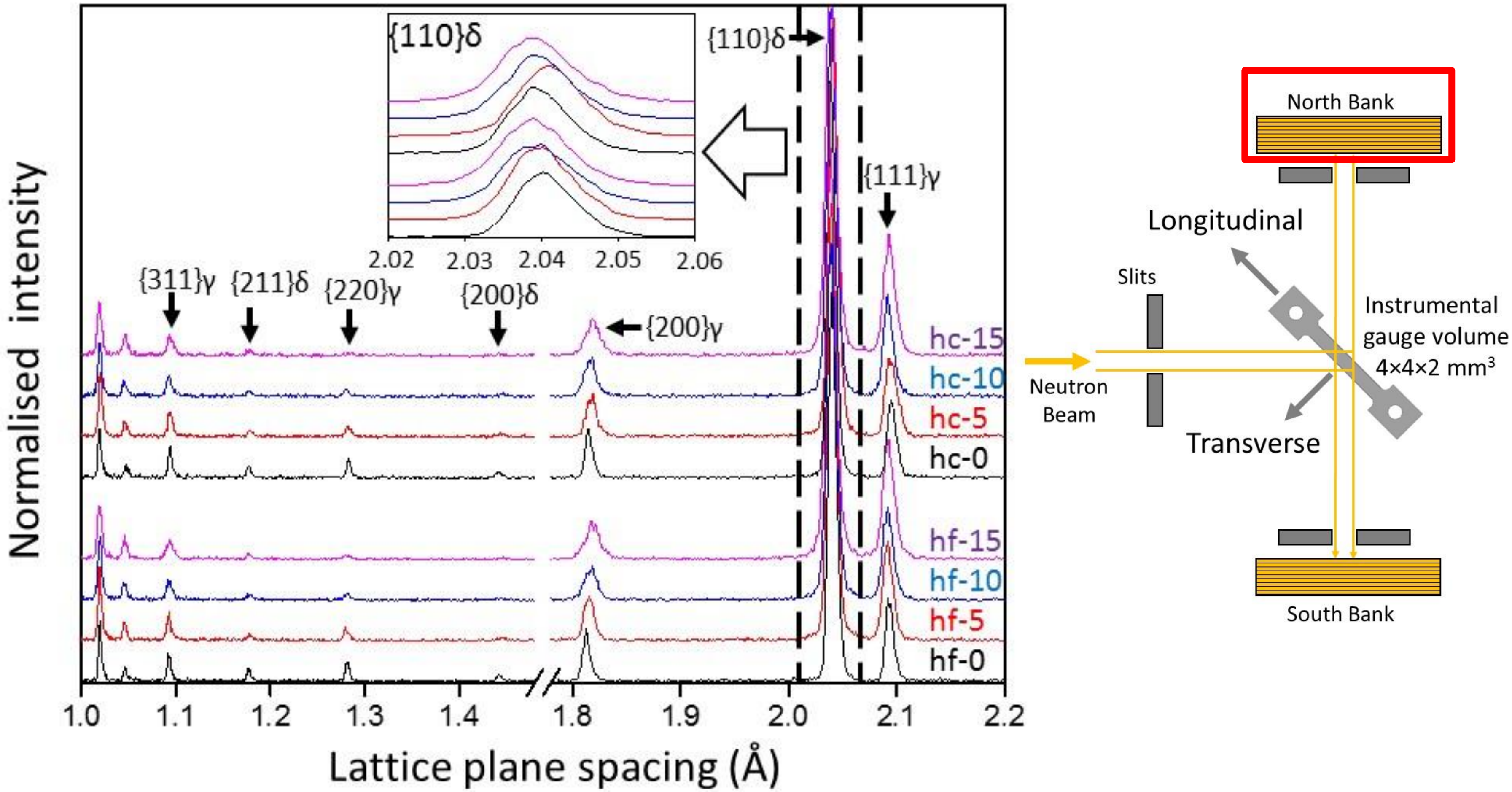
Dislocation density equals (ρ_b+10^{12}) m^{-2}

FWHM stands for full width at half maximum.

Results and discussion

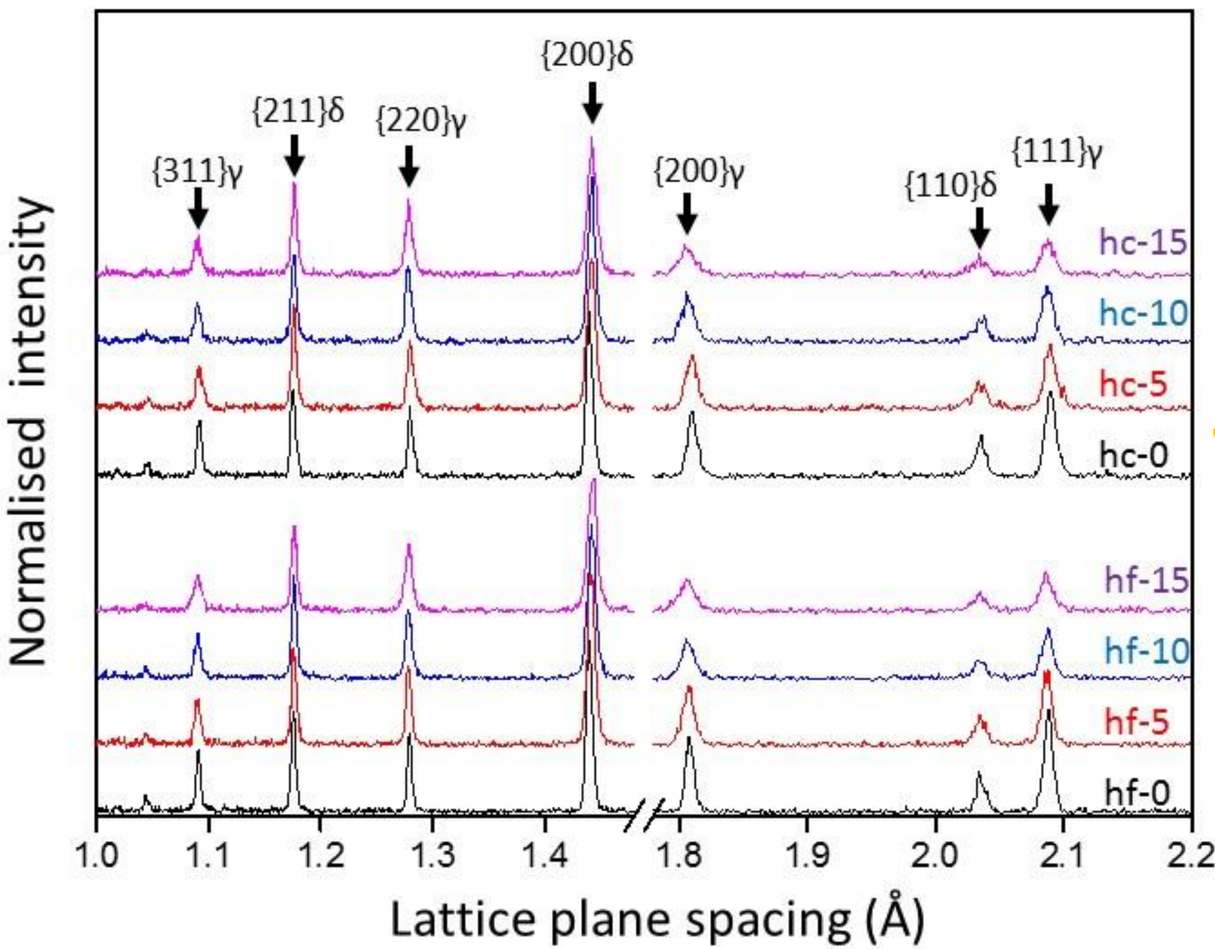


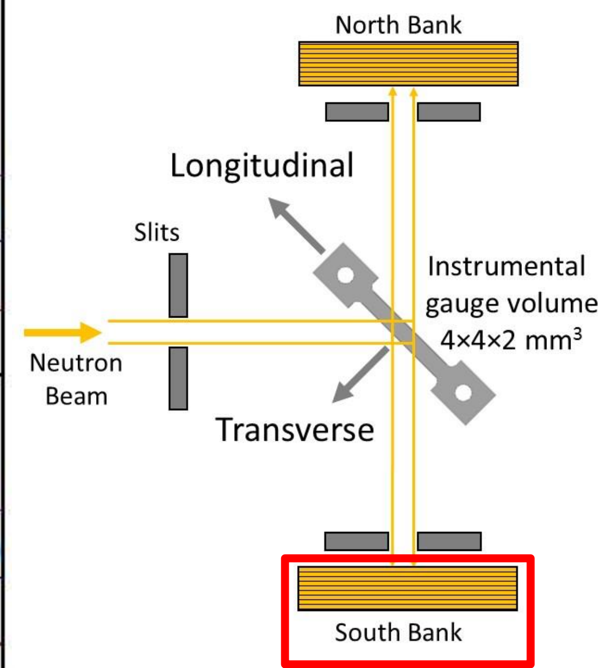
Spectrums of neutron diffraction-Longitudinal



abbreviation of hc and hf strand for hydrogen charged and hydrogen free, respectively.

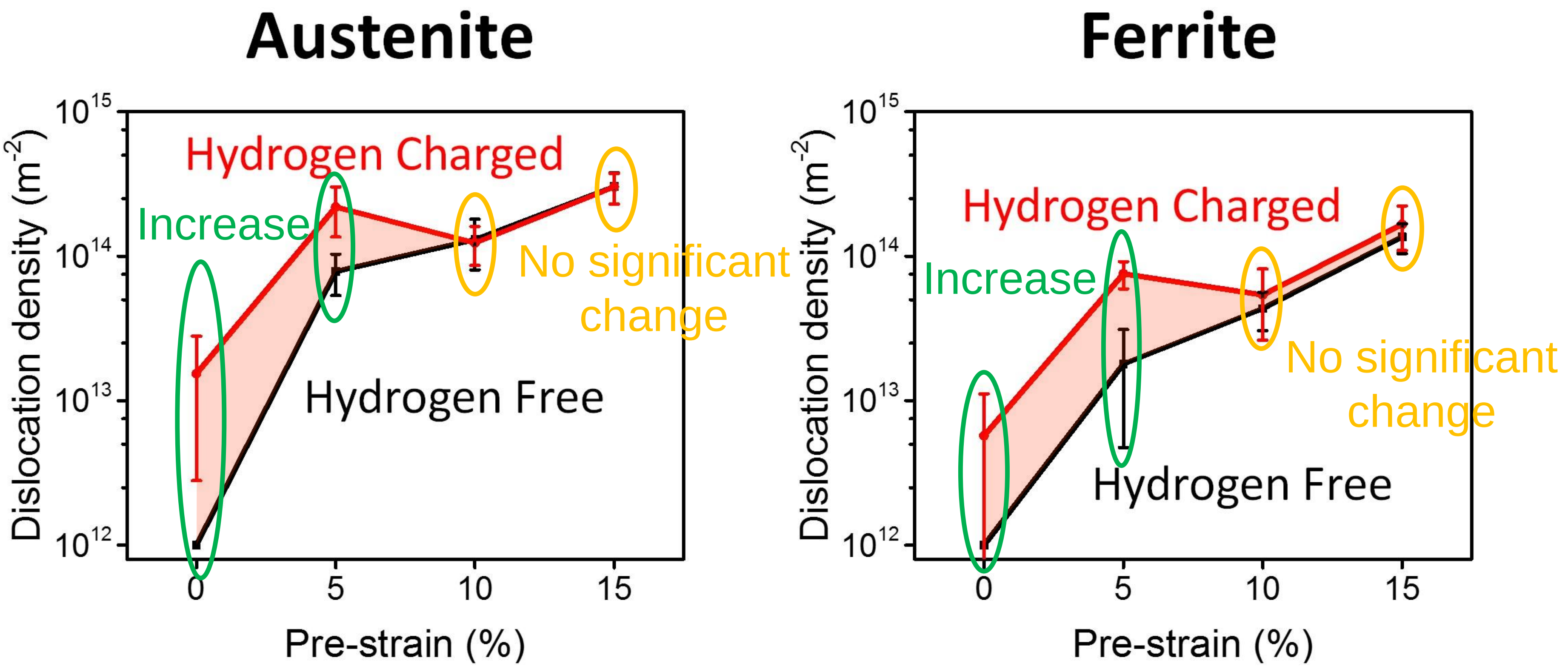
Spectrums of neutron diffraction-Transverse





abbreviation of hc and hf strand for hydrogen charged and hydrogen free, respectively.

Measured dislocation density



Total dislocation density was calculated by averaging the measured values of dislocation density from longitudinal and transverse directions.

Stored dislocation density

The stored dislocation density is a **balance** of **athermal storage of dislocation** and static **recovery**

Dislocation multiplication Dislocation annihilation

$$\frac{d_\rho}{d_\varepsilon} = \frac{d_{\rho^+}}{d\varepsilon} + \frac{d_{\rho^-}}{d\varepsilon}$$

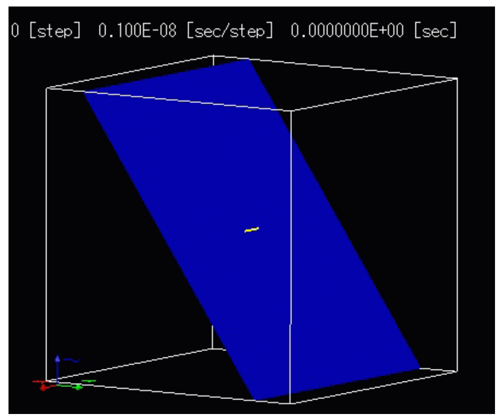
Where the term $d_{\rho^+}/d\varepsilon$ is dislocation generation and term $d_{\rho^-}/d\varepsilon$ is dislocation annihilation.

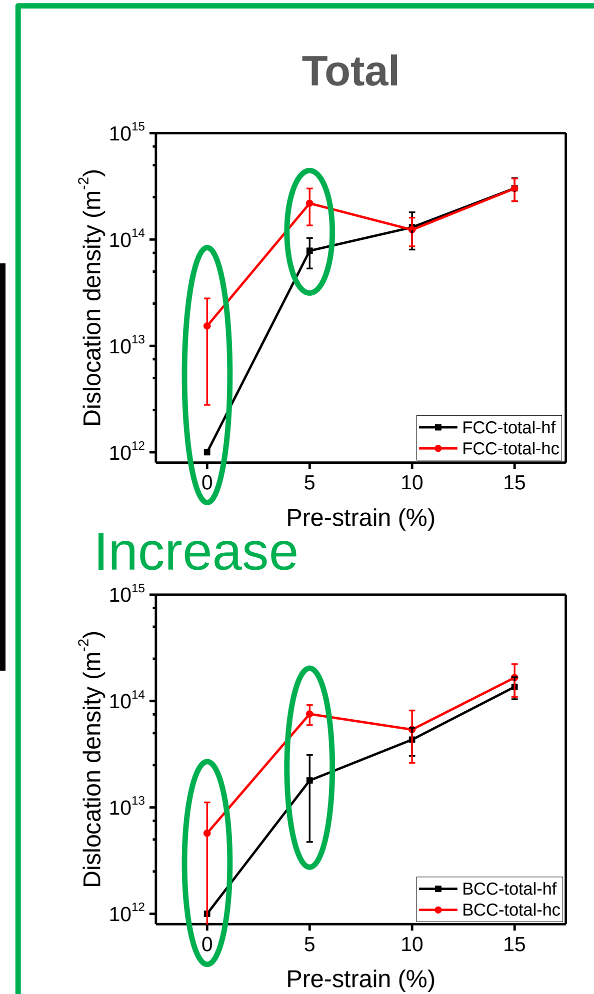
Dislocation multiplication

In SDSS, Frank-read-type source is suggested to account for dislocation density multiplication. The critical shear stress required to activate the source is:

$$\tau = u\frac{2Gb}{l_d}$$

Unit of dislocation line energy is reduced with hydrogen solutes presentation





Where G is the shear modulus, l_d is the segment length and u is the unit of dislocation line energy.

Animation adopted from: *https://en.wikipedia.org/wiki/Frank-Read_source*

Dislocation annihilation

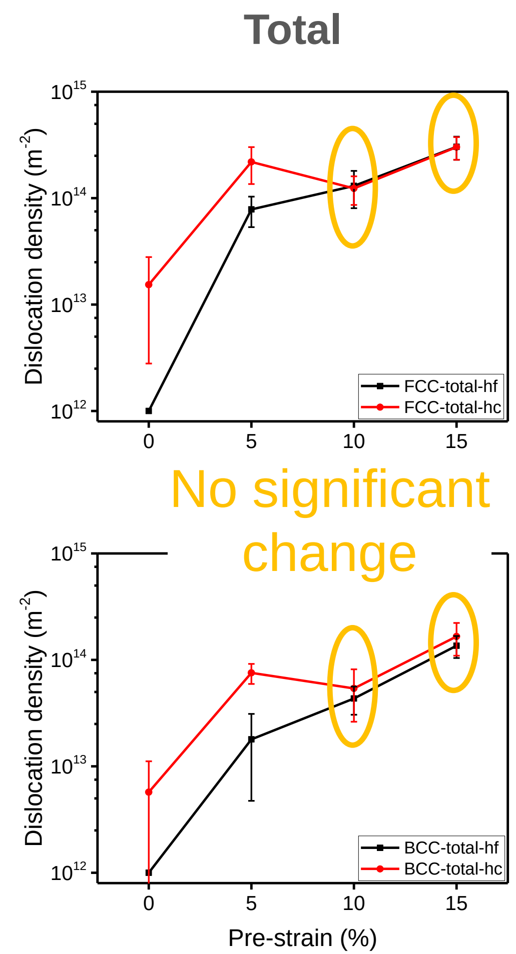
Kocks and Mecking proposed a systematic model to evaluate dislocation density within conditions of varying temperature and strain. If we only consider the strain effect, the net storage rate of dislocation can be written as:

multiplication annihilation

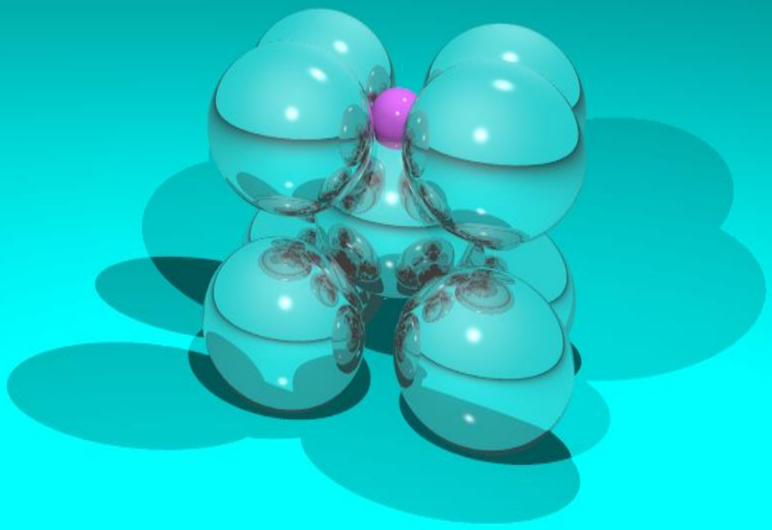
$$\frac{d_{\rho}}{d_{\varepsilon}} = M(k_1\sqrt{\rho} - k_2\rho)$$

where M is Taylor's factor; k_1 is storage constant; k_2 is dynamic recovery constant which is proportional to the critical dislocation annihilation distance. Both k_1 and k_2 are positive values.

As $\boldsymbol{k_2\rho}$ **possess higher order of term** $\boldsymbol{\rho}$ **than** $\boldsymbol{k_1\sqrt{\rho}}$**,** with increasing strain, the increasing of dislocation density tend to slow down and finally achieve a **dislocation saturated status**. In present study, hydrogen gave a raise of dislocation density in unstrained and 5% pre-strained samples while fail to increase dislocation density in 10% and 15% pre-strained samples which may owning to dislocation saturation.



Conclusions



Conclusion

- Neutron diffraction is an effective probing method to study the dislocation density in bulk materials.
- Hydrogen induced dislocation multiplication is a function of pre-strain, where dislocation density increase manifestly in samples with less than 5% pre-strain. Such dislocation multiplication is impeded when pre-strain reaches 10%.

References

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7. U.F. Kocks, H. Mecking, Physics and phenomenology of strain hardening: the FCC case, Prog. Mater. Sci. 48(3) (2003) 171-273.

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Thank you