

Incoherent Neutron Scattering Measurements of Hydrogen-Charged Zircaloy-4

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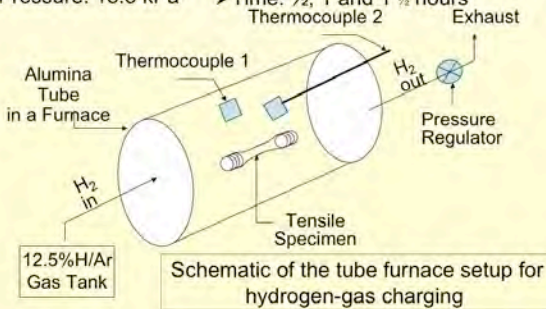
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(1) Abstract

Qualitative and quantitative phase measurements were conducted on Zircaloy-4 round bars using neutron scattering techniques. The mapping through the thickness of the specimens using neutron diffraction showed the presence of the face-centered-cubic delta zirconium hydride (δ -ZrH₂) phase on the surface. To determine the relative amount of hydrogen in the Zircaloy-4 samples, the increase of the incoherent scattering with the hydrogen content was calibrated using standard samples for which the hydrogen content was known.

(2) Hydrogen Charging

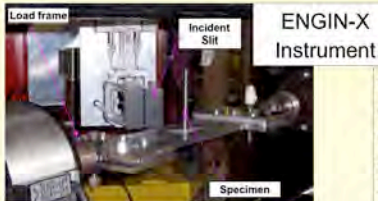
- H gas (12.5% H in Ar)
- Promoter: Ni layer, 0.15 μ m
- Temperature: 430 °C
- Pressure: 13.8 kPa
- Time: 1/2, 1 and 1 1/2 hours



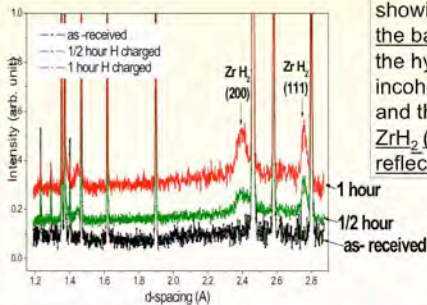
(3) Neutron Scattering Measurements

(A) Phase Identification

ISIS, Rutherford Appleton Laboratory, UK



Comparison of 3 diffraction patterns, corresponding to the as-received and hydrogenated specimens (1/2 and 1 hour charged), showing increases in the background due to the hydrogen incoherent scattering and the presence of δ -ZrH₂ (111) and (200) reflections.

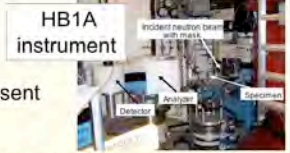


Acknowledgement

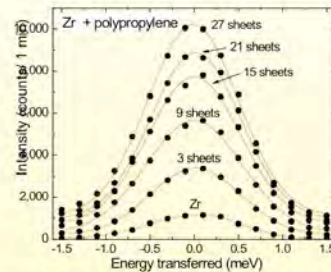
- National Science Foundation (NSF): International Materials Institutes (IMI) Program (DMR-0231320), managed by Dr. C. Huber.
- Tennessee Advanced Material Laboratory (TAML) Fellowship Program managed by Prof. W. Plummer.
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(B) Hydrogen Content in the Bulk, HFIR, Oak Ridge National Lab.

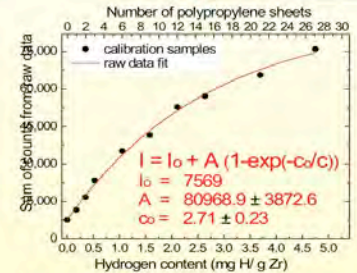
- Energy scans range : -1.5 meV to 1.5 meV,
- Step: 0.2 meV , 60 seconds per point.
- $2\theta = 62$ deg.
- Flat background such that no coherent scattering is present



Calibration

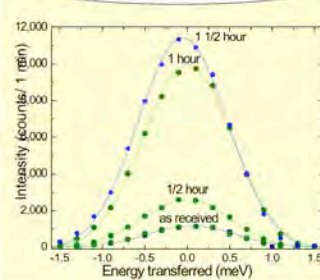


Incoherent scattering of calibration samples with polypropylene sheets

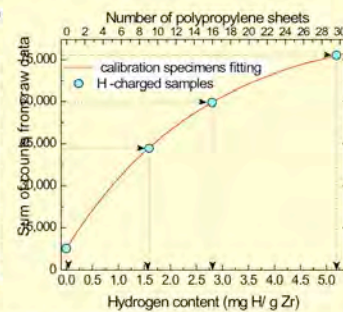


Fit (red line) of the experimental data for the polypropylene sets

Hydrogenated samples



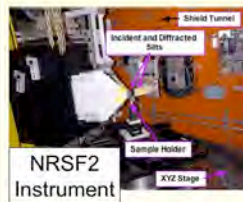
Incoherent scattering increases with the hydrogen content in the hydrogen-charged Zr-4 specimens



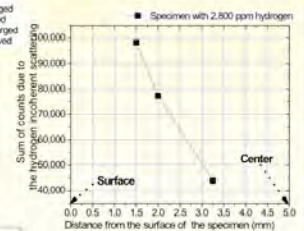
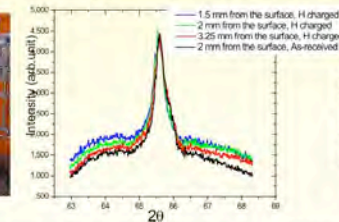
Charging time (hour)	Hydrogen content (ppm)
1/2	1,600
1	2,800
1 1/2	5,200

Estimation of the hydrogen content for the samples charged with hydrogen

(C) Mapping of Hydrogen Content Through the Thickness of a Specimen with 2,800 ppm Hydrogen, HFIR, ORNL



Comparison of 3 diffraction patterns, measured at different depths in the specimen with 2,800 ppm hydrogen, to the as-received specimen (no hydrogen), showing increases in the background due to the hydrogen incoherent scattering



Through-thickness variation of the hydrogen content

(4) Summary

- ✓ A fcc δ -ZrH₂ phase was identified on the surface of Zr-4 bars charged with hydrogen gas for different amounts of time.
- ✓ Incoherent scattering of hydrogen caused increases in the background of the diffraction profiles, which allowed the quantification of the average hydrogen content in the bulk.
- ✓ Through thickness mapping of a hydrogen charged specimen showed that the distribution of the hydrogen within the Zr-4 round bar can be quantitatively mapped.