

Local Hydrogen Concentration Determination in Zircaloy Cladding Material

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Local Hydrogen Concentration Determination in Zircaloy Cladding Material

Method: general overview

Sample preparation

cutting
grinding
polishing

Scanning electron microscopy (SEM)

Back scattering mode (Robinson detector)

Digital image analysis

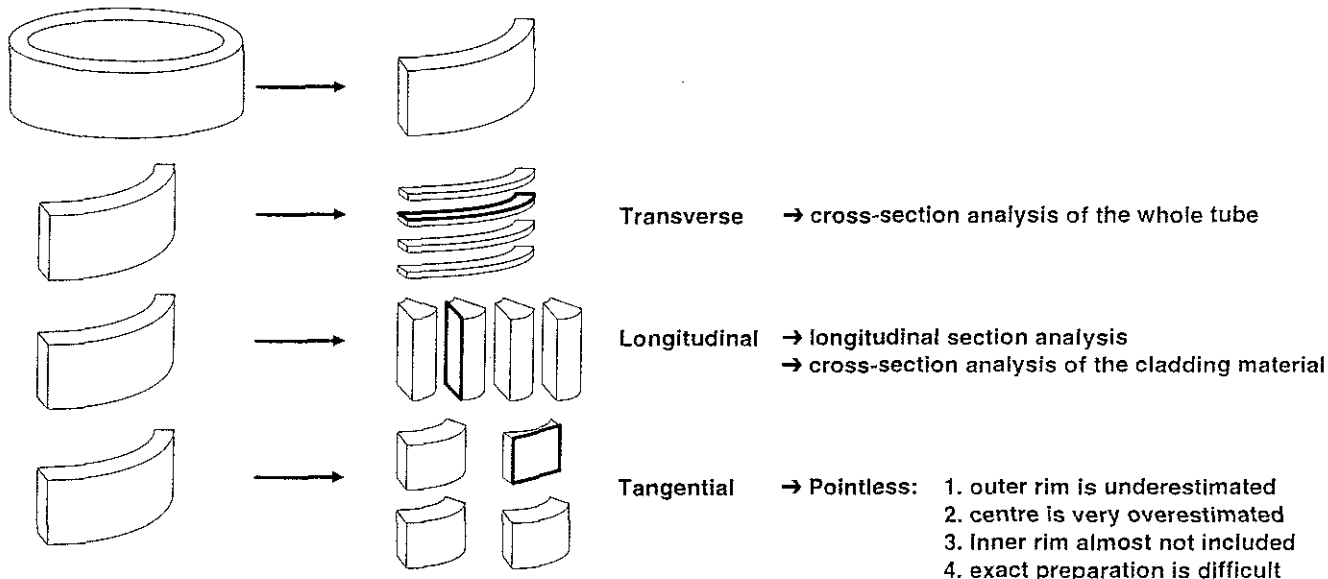
image processing
image analysis

Calculation

calculation
statistics
(graphical) presentation

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Sample preparation (1)



Local Hydrogen Concentration Determination in Zircaloy Cladding Material

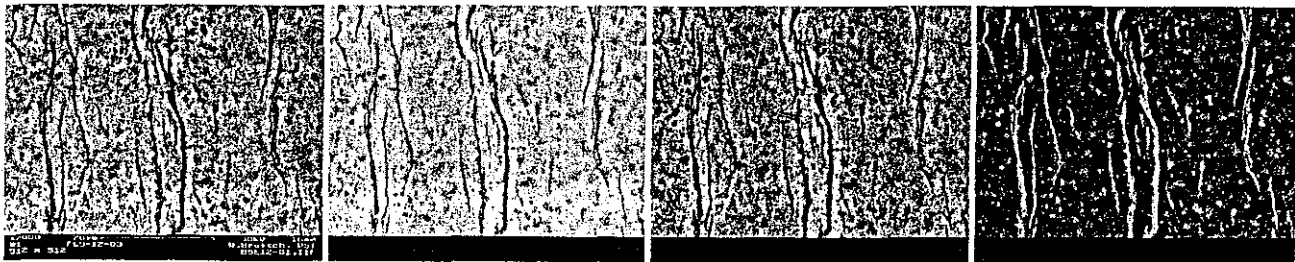
Sample preparation (2)

Grinding	Grid 500	until plane surface
	Grid 2400	50 μm
	Grid 4000	20 μm
Polishing	OP-S (SiO_2 coll.)	15 Minutes
		no chemicals (e.g. HF, H_2O_2 etc.)
		→ pure mechanical polish
		→ almost no topographic effects (pure material contrast)
		→ almost no oxidation on surface (conductivity)
Cleaning	Ultrasonic bath, soap water, then rinse with pure water	
	Ultrasonic bath, acetone	

Local Hydrogen Concentration Determination in Zircaloy Cladding Material Scanning electron microscopy (SEM)

SEM:	Zeiss DSM 962
Detector:	Robinson Detector
High Voltage:	30 kV
Probe Current:	0.3 nA
Contrast setting:	maximum
Magnification:	2000 x

Local Hydrogen Concentration Determination in Zircaloy Cladding Material Digital Image Analysis

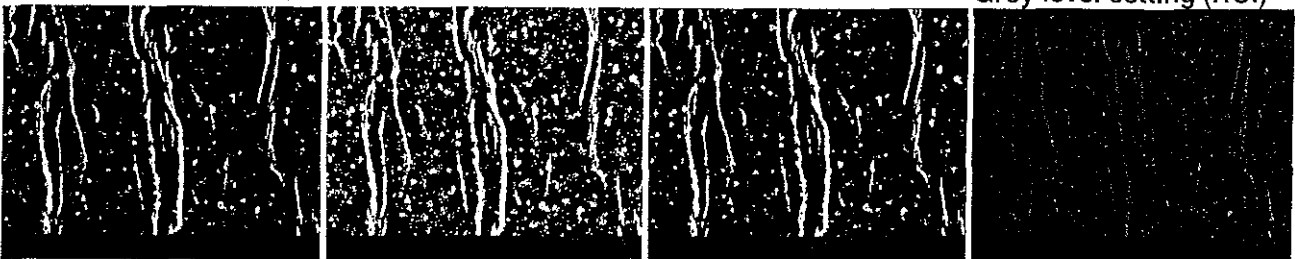


Original BSE-Image

Shading Correction

Contrast enhancing

Inverting grey values /
Grey level setting (ROI)



Create a binary image

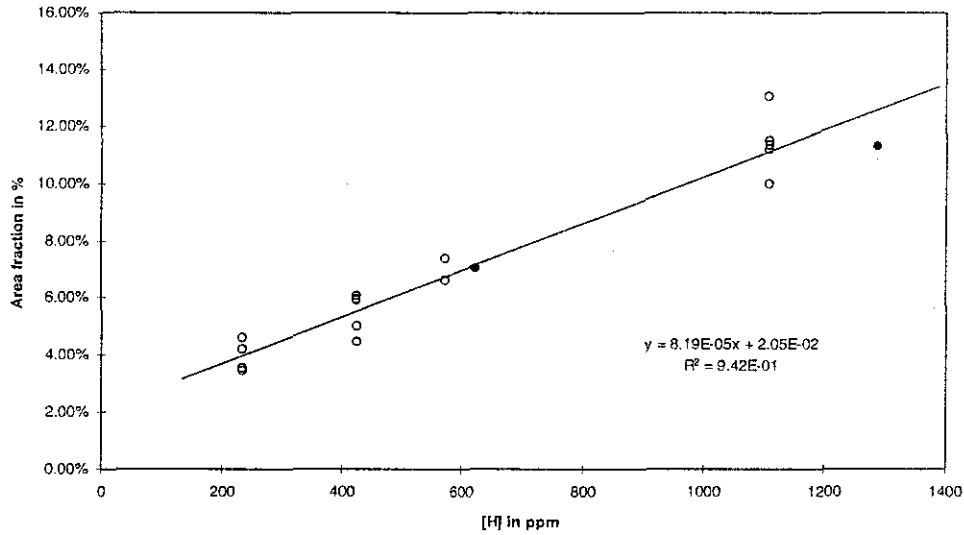
Dilatation
(Close Particles)

Erosion
(reduce the Oversize)

Detection of the
Regions of interest

Local Hydrogen Concentration Determination in Zircaloy Cladding Material Calibration

Correlation of the hydrogen area fraction vs. standard hydrogen concentration



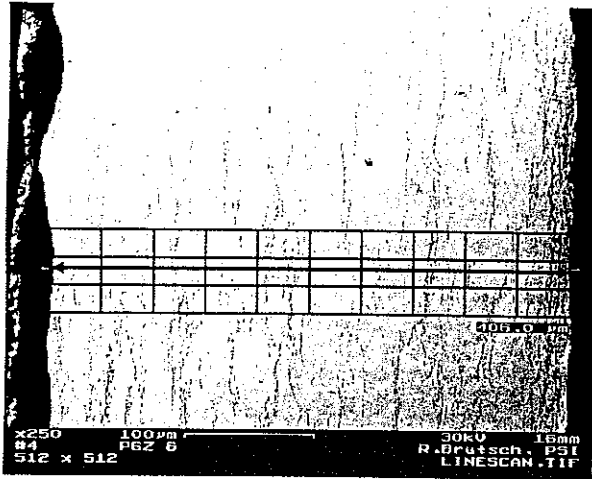
Local Hydrogen Concentration Determination in Zircaloy Cladding Material Comparison of Precision / Resolution

	Hot Extraction (LECO)	SEM and Digital Image Analysis
Reproducibility	$\pm 2 \%$	$\pm 12 \%$
Precision	$\pm 4 \%$	$\pm 24 \%$
Resolution	65 mg \approx $\frac{1}{4}$ of a 3.6 mm Ring	45 x 58 $\mu\text{m}^2 = 170 \text{ ng}$

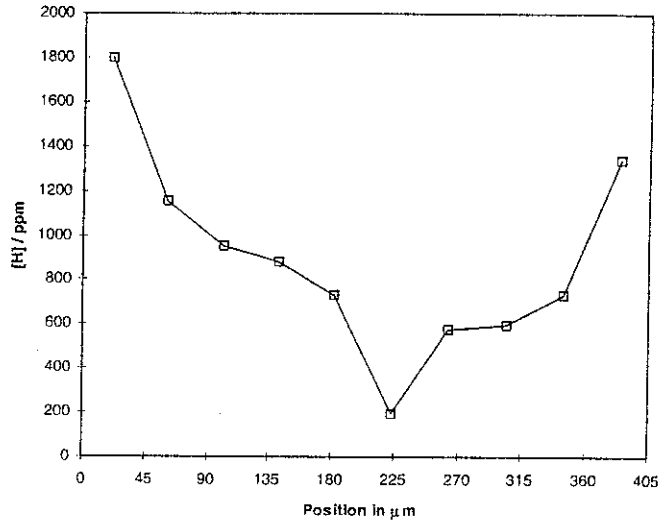
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Quantitative line scan

BSE-Image



Line scan



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Possible applications

