EPMA vs. LA-ICP MS
case study of Si in AL-5052

M. Verwerft, S. Van Winckel, J. Van de Velde and L. Vandevelde
SCK•CEN
Boeretang, 200 B-2400 Mol, Belgium
Si in Al-5052: EPMA vs LA-ICP MS

- Problem definition
- Practical restraints
- Results and error discussion
- Lessons learned...
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Refurbishment of BR2 MTR: how about the fracture toughness evolution?

No modelling insight, no surveillance, little data from other MTR's, (incorrect) estimate of fluence: close to accelerated degradation threshold

SAMPLING of BR2 'shroud'
Figure 1.1.2.

General view of the BR2 reactor
BRZ Vessel
FIG. 6 Calculated thermal neutron fluences ($n_v$, $t$) in the BR2 vessel (situation mid-1995)
FIG. 10 Thermal fluence ($\nu_0 t$) in the BR2 vessel at mid-1995 as calculated times fluence ratio indicated in Fig. 8
Fig. 2.4 Cutting scheme of BR2 Aluminium shroud.

PLATE U: Weld + Base material

- Pieces for microstructure analysis, chemical analysis and dosimetry
Fracture toughness evaluation

- Charpy-V notch
- precracked Charpy-V
- smooth tensile
- notched tensile

Chemical characterisation

- EPMA
- LA-ICP MS
- ICP AES
- GD MS
Si in Al-5052: EPMA vs LA-ICP MS

\[ \beta^- \]

Al-27 \((n,\gamma)\) Al-28 \(\rightarrow\) Si-28 \((n,\gamma)\) Si-29 \((n,\gamma)\) Si-30 ...

234 mb \hspace{1cm} 207 mb \hspace{1cm} 120 mb

\[ 1.0 \times 10^{22} \text{ n/cm}^2 \rightarrow 0.234 \text{ at}\% \text{ Si} \]

Gradual transition from 5xxx series to 6xxx alloy
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- Active material
- Limited amount available
- Large number of samples, with slightly different microstructure
- Silicon in aluminium matrix ...
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EPMA

- Si signal is most efficiently absorbed by Al
- Transmutation produced Si: quasi-homogeneous
- Low concentrations impose long acquisition times

LA-ICP MS

- Si signal too weak for direct comparison with Al signal
- Quadrupole MS yields high background below Si signal calibration is based on standards with highly different structure (sampling!?)
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- LA-ICP MS
  - direct Si$^{28}$/Al$^{27}$ impossible: Si$^{28}$/Mg$^{25}$ (NN & CO interference); Mg$^{25}$/Al$^{27}$: different lens settings
  - Mass Bias Factor (MBF)
  - Sensitivity Factor (SF): from standards which differ from a metallurgical point of view (LA sampling !!)
  - "long" campaign: e.g. torch exchange,...
  - ...

[...]

[...]

[...]
Si in Al-5052: EPMA vs LA-ICP MS

EPMA

- ZAF-correction factors
- inhomogeneous material
- Chemical shift of Si, K$_{\alpha}$ line energy
- sample preparation induced artifacts
- ...


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Histogram Analysis

Frequency

Si concentration (wt%)
Produced Si as a function of specimen position
Produced Si as a function of specimen position
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- Four techniques ----> four results
- Repeatability is much better than accuracy
- Honest error estimation often lacks
- Validation is necessary, but difficult for non-standard work
- Call for a R-R definition on solid state analyses