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

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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'

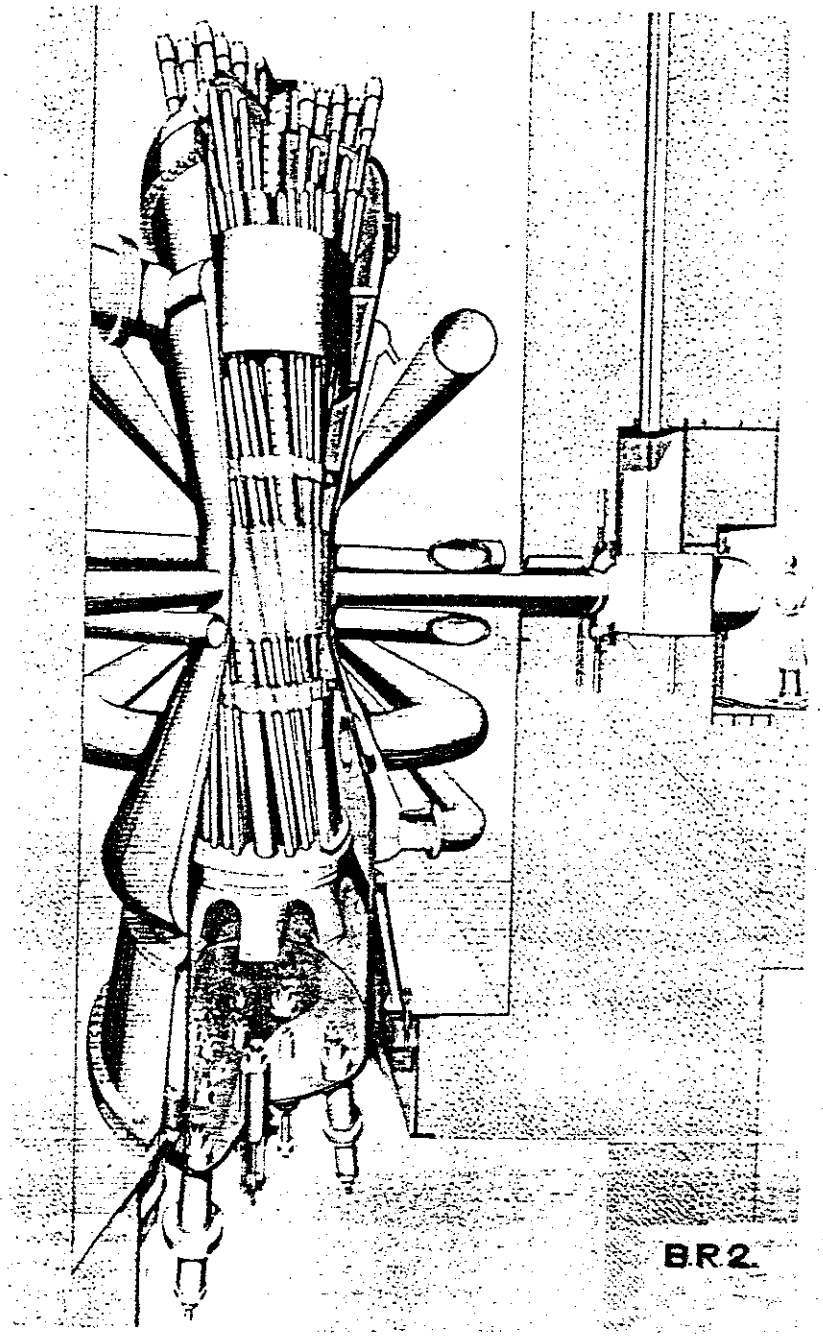


Figure 1.1.2.

General view of the BR2 reactor

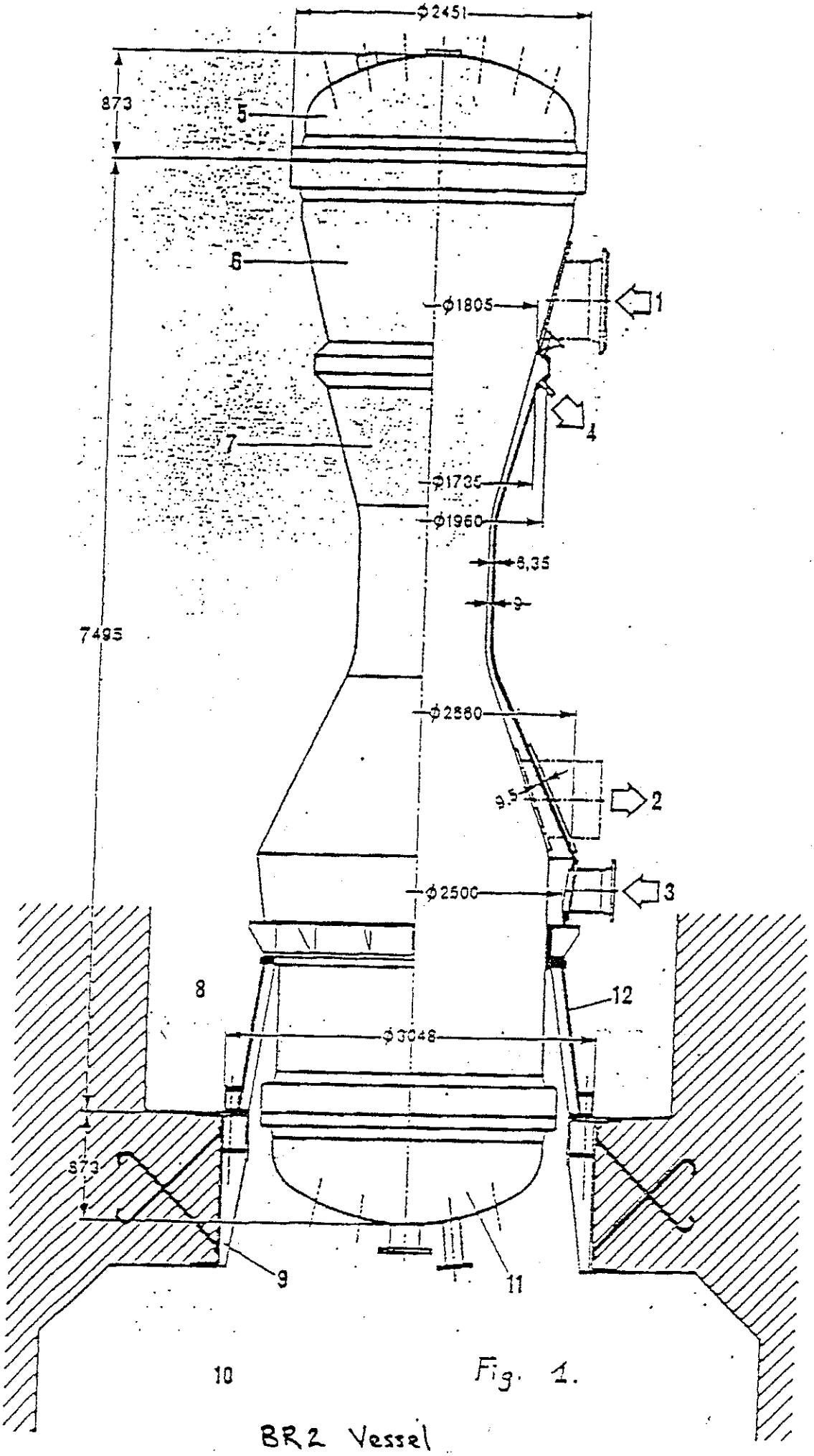


Fig. 1.

BR2 Vessel

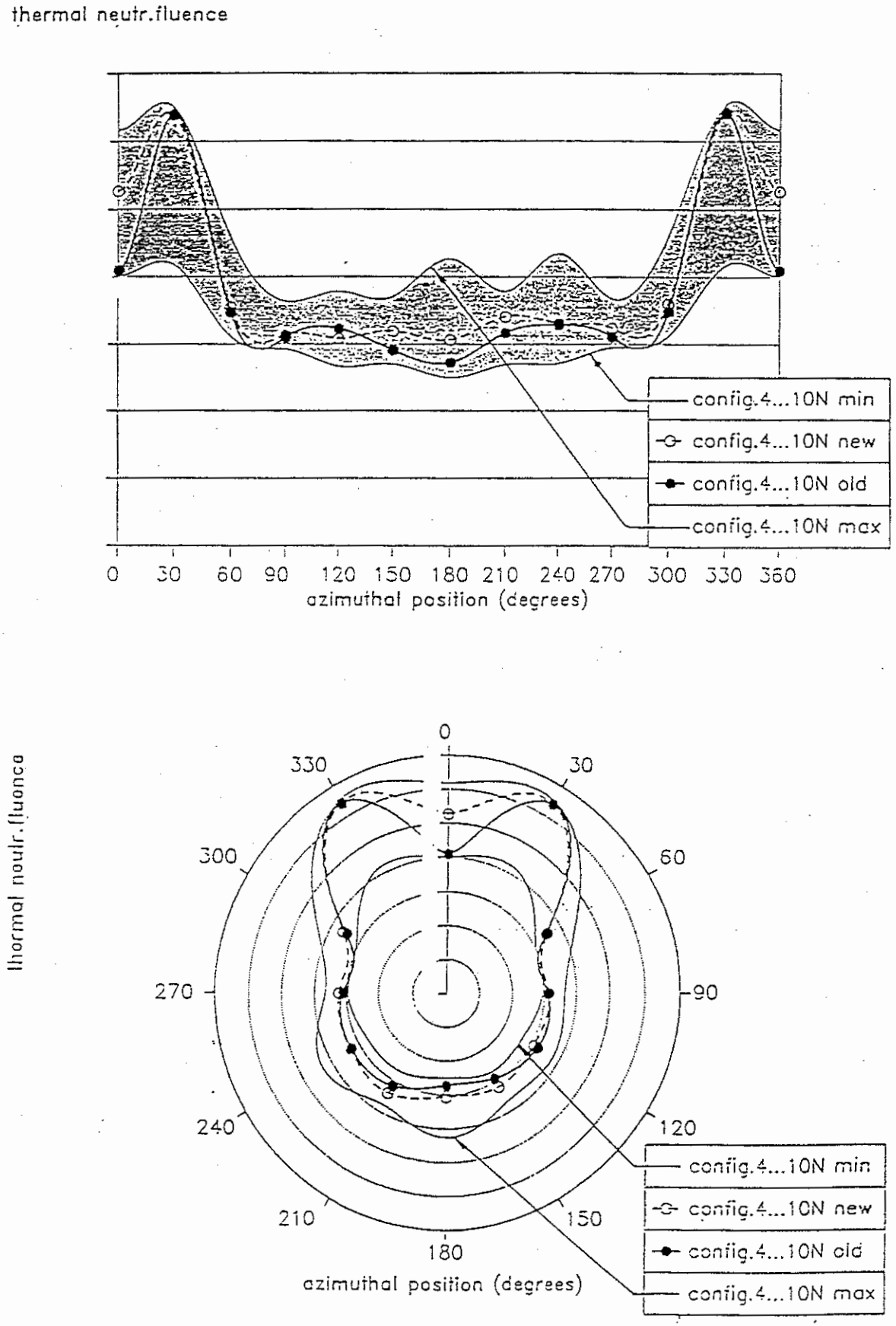


FIG.6 Calculated thermal neutron fluences (nv_0 t) in the BR2 vessel (situation mid-1995)

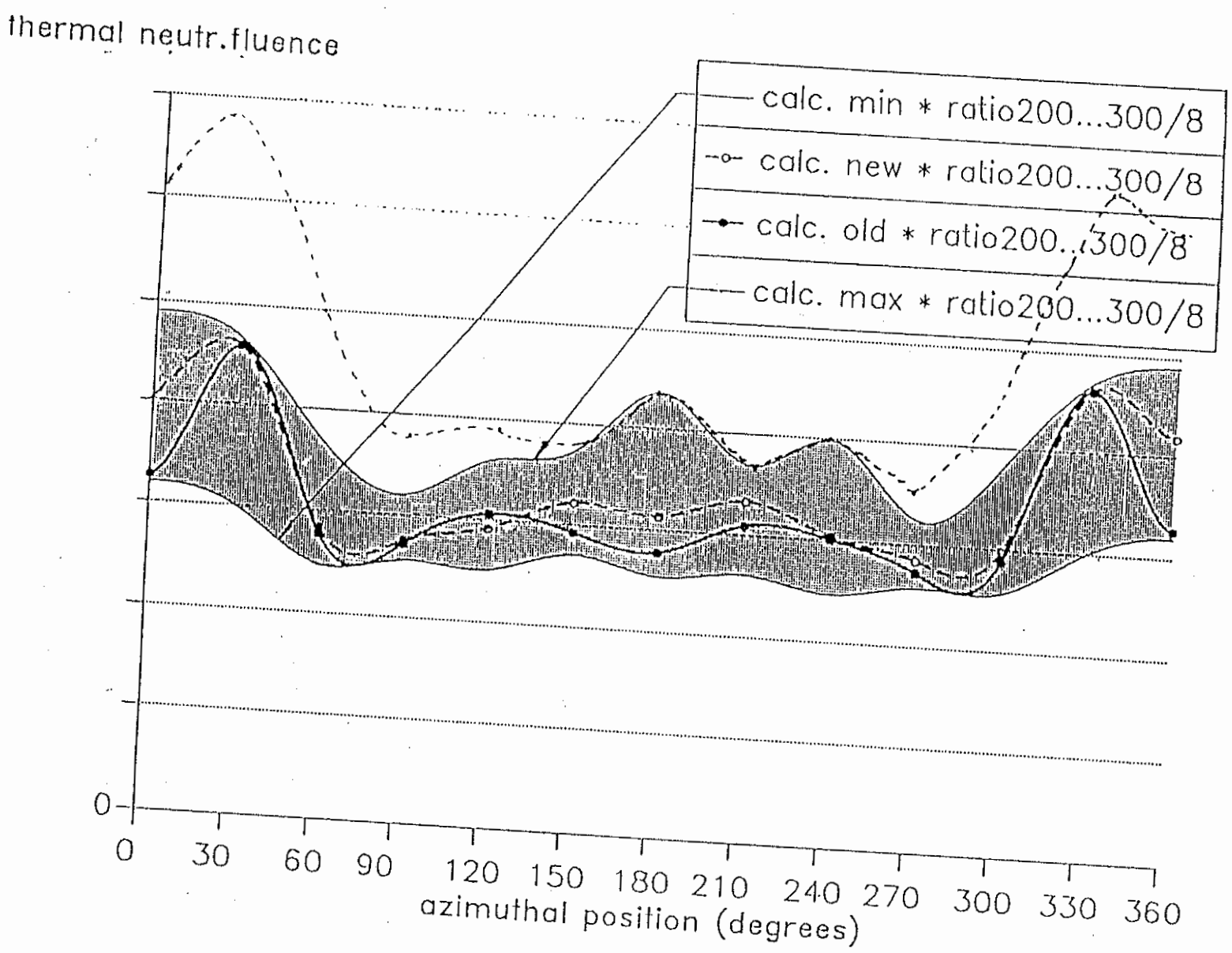
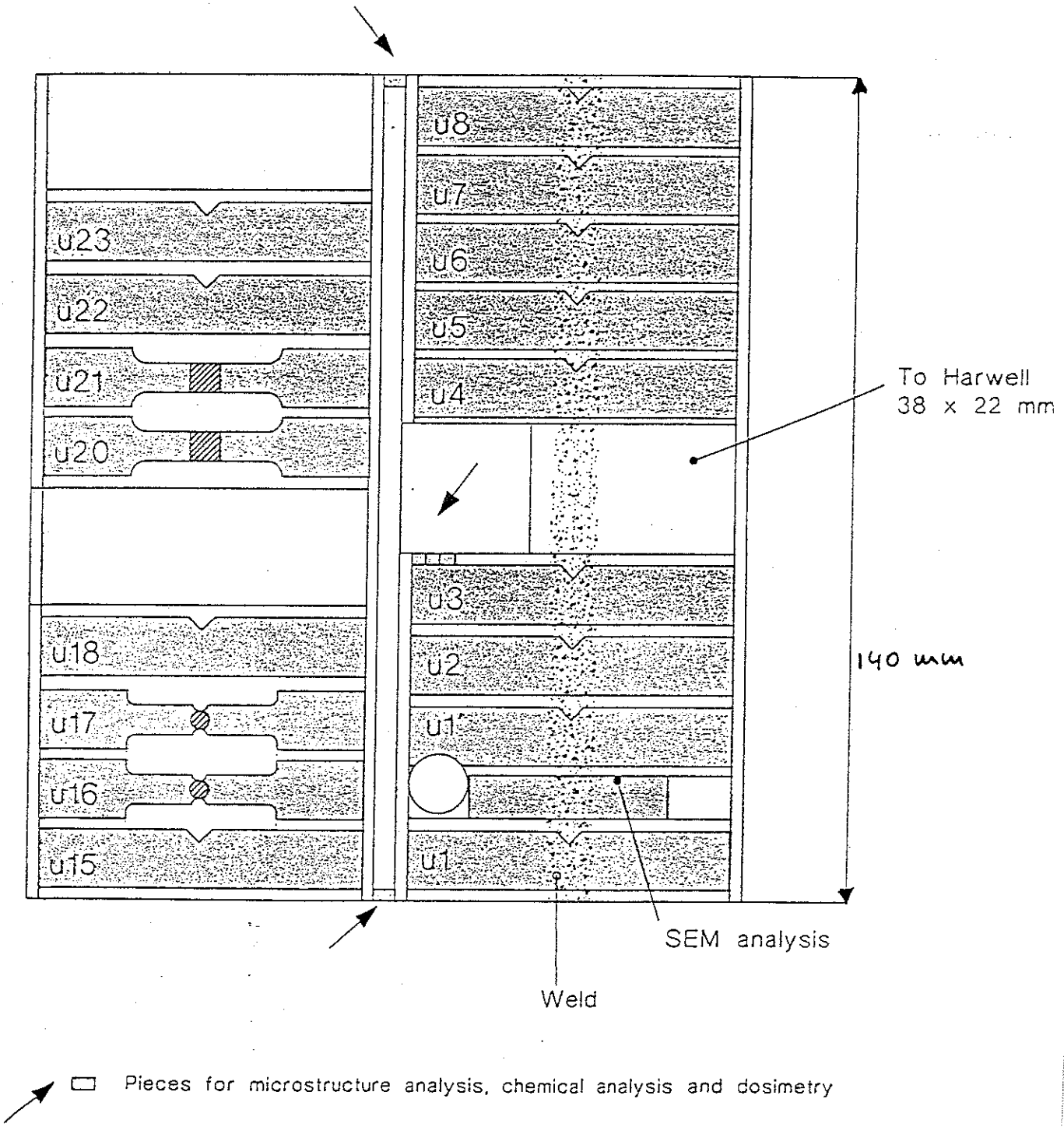


FIG.10 Thermal fluence (nv_0 t) in the BR2 vessel at mid-1995 as calculated x fluence ratio indicated in Fig. 8

Fig. 2.4 Cutting scheme of BR2 Aluminium shroud.

PLATE U : Weld + Base material



Pieces for microstructure analysis, chemical analysis and dosimetry

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

- 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

$$\text{Al-27 } (n,\gamma) \text{ Al-28} \xrightarrow{\beta^-} \text{Si-28 } (n,\gamma) \text{ Si-29 } (n,\gamma) \text{ Si-30 } \ldots$$

234 mb 207 mb 120 mb

$1.0\ 10^{22}$ n/cm² ----> 0.234 at% Si

Gradual transition from 5xxx series to 6xxx alloy

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

- Active material
- Limited amount available
- Large number of samples, with slightly different microstructure
- Silicon in aluminium matrix ...

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

- EPMA
  - Si signal is most efficiently absorbed by Al
  - transmutation produced Si:quasi- **homogeneous**
  - alloy Si: **inhomogeneous**
  - 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!!)

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

- LA-ICP MS
  - direct Si28/Al27 impossible: Si28/Mg25 (NN & CO interference); Mg25/Al27: 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,...
  - ...

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

- EPMA
  - ZAF-correction factors
  - inhomogeneous material
  - Chemical shift of Si,K_α line energy
  - sample preparation induced artifacts
  - ...

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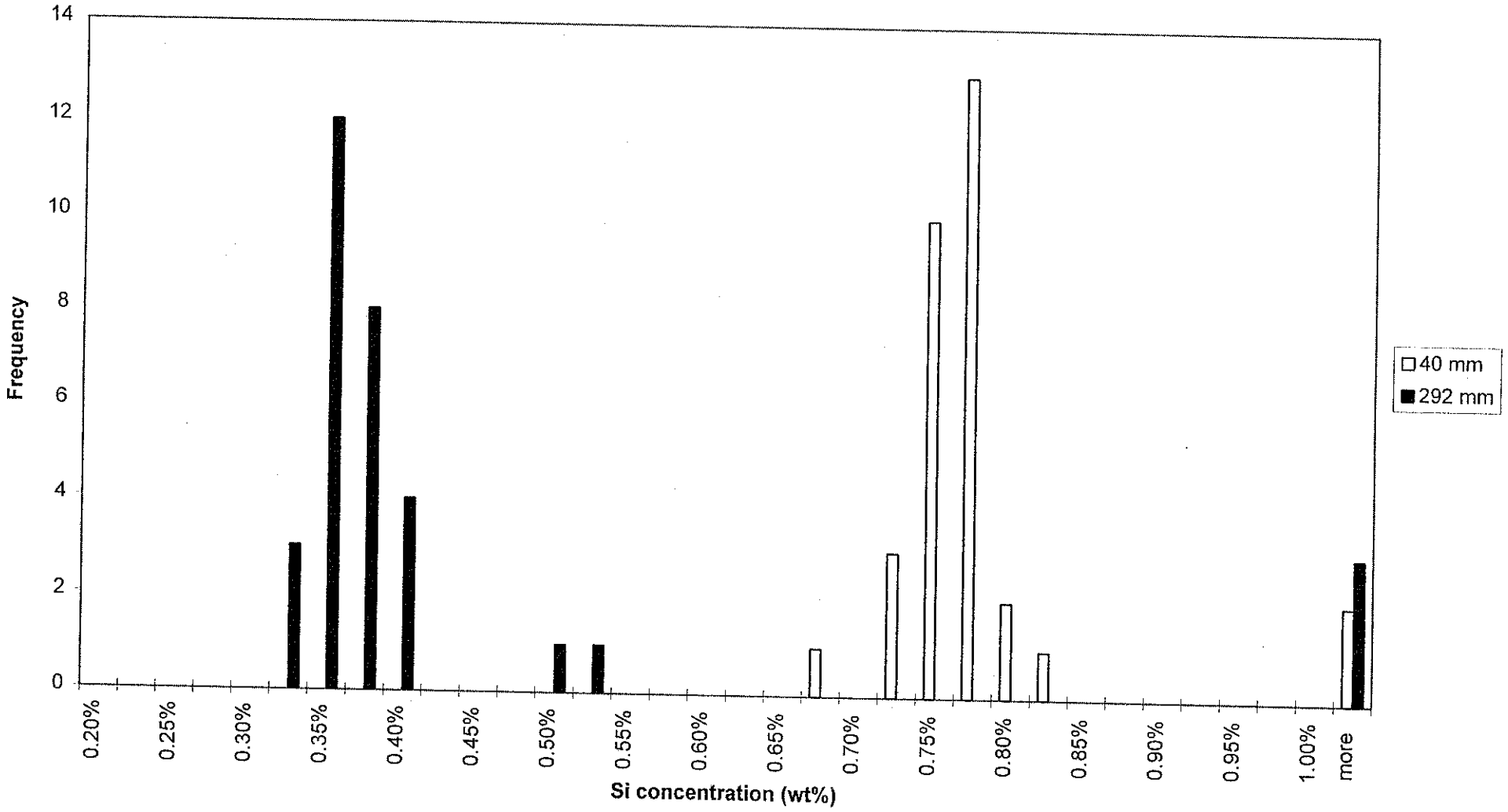
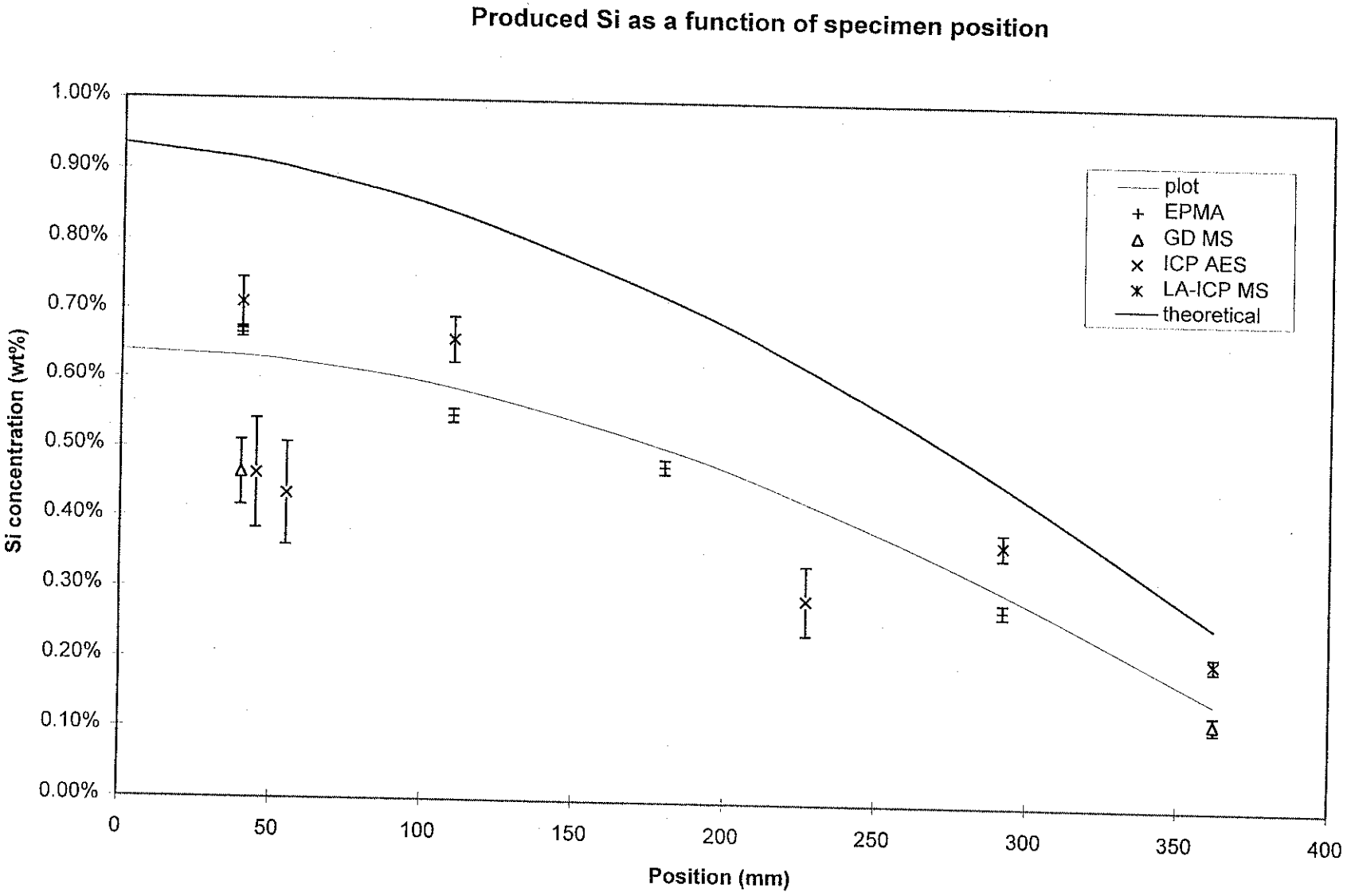
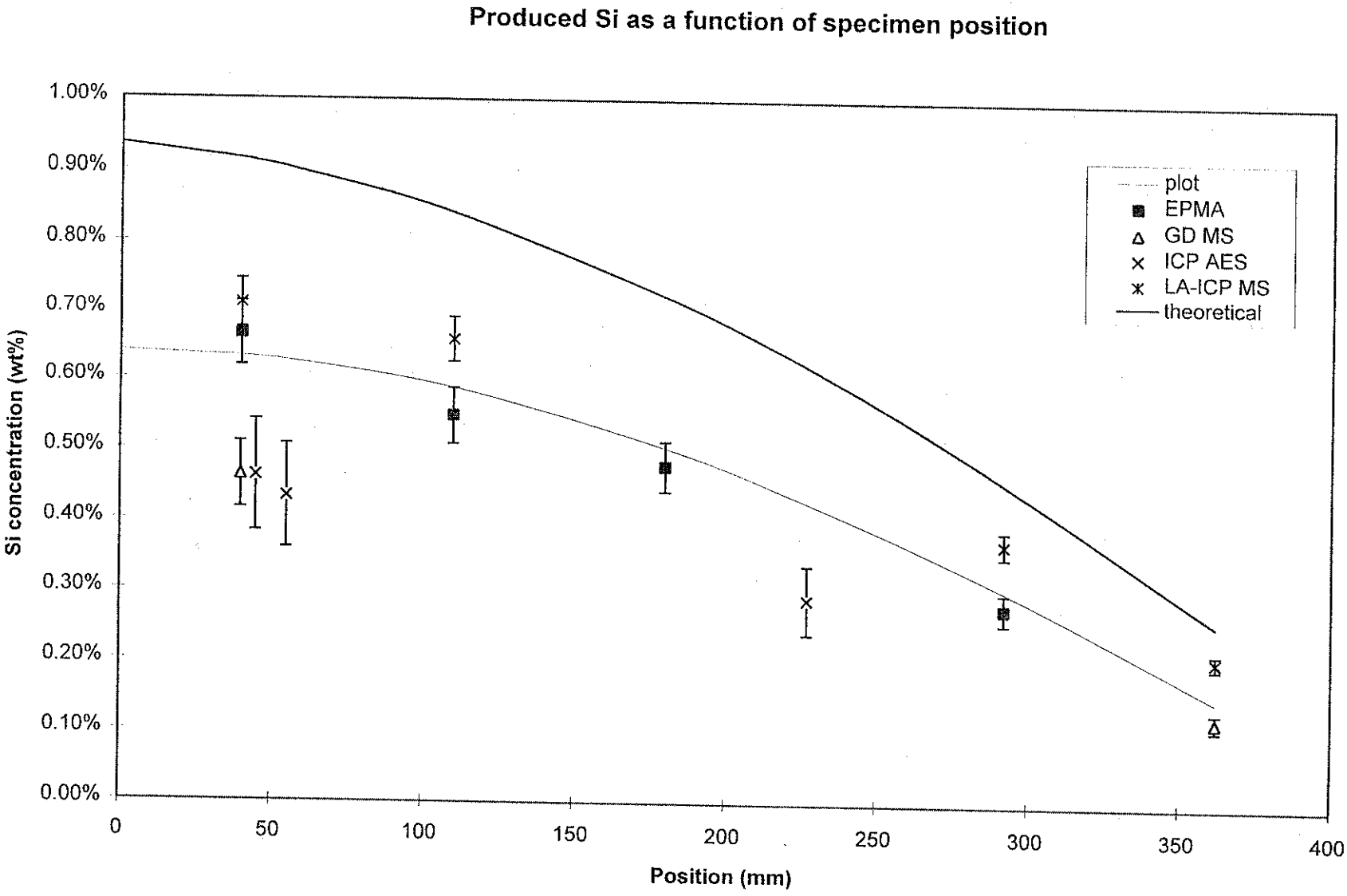
Histogram Analysis




Produced Si as a function of specimen position
Si concentration (wt%)
1.00%
0.90%
0.80%
0.70%
0.60%
0.50%
0.40%
0.30%
0.20%
0.10%
0.00%
0
50
100
150
200
250
300
350
400
Position (mm)
plot
EPMA
GD MS
ICP AES
LA-ICP MS
theoretical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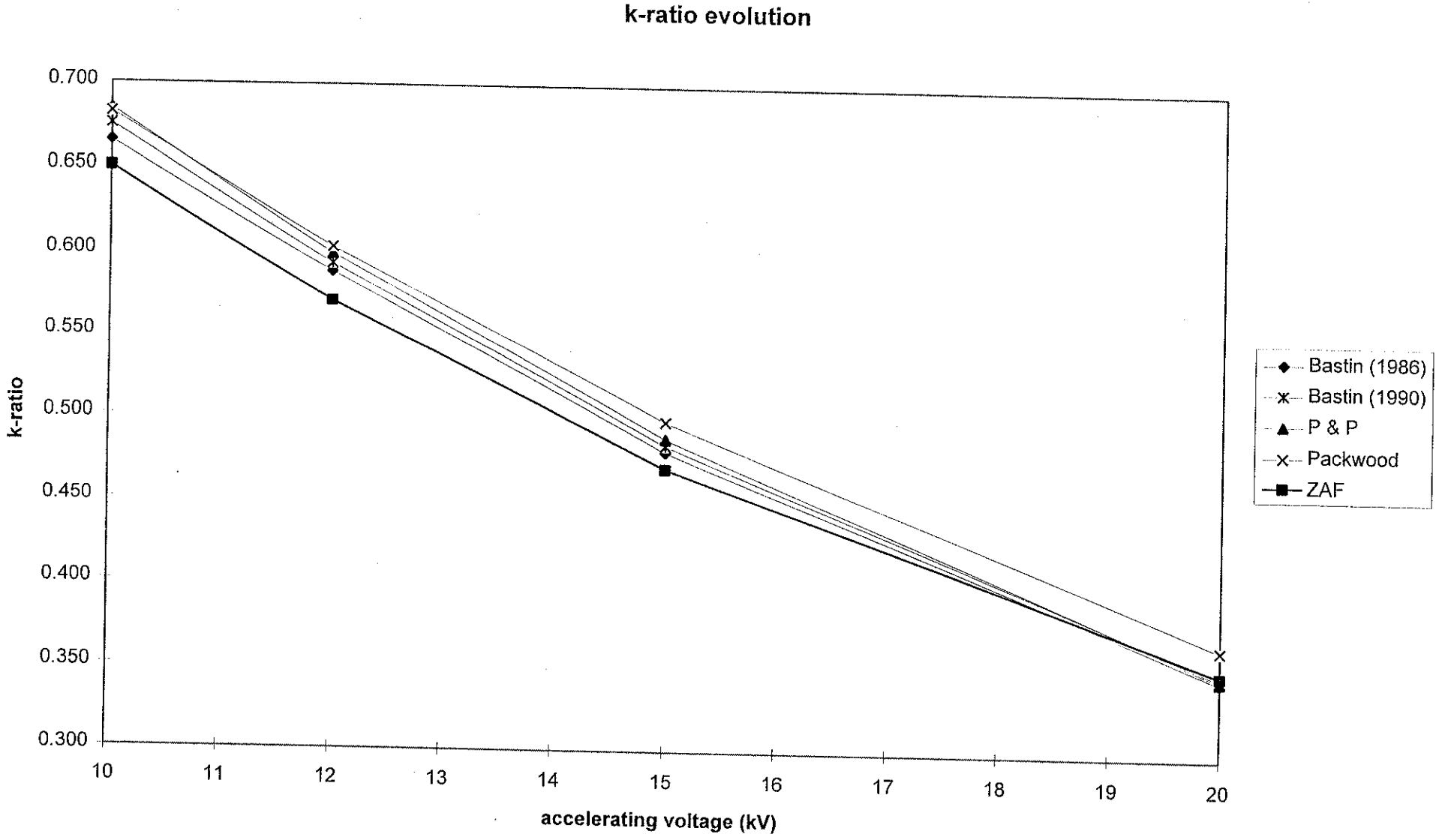
k-ratio evolution
0.700
0.650
0.600
0.550
0.500
0.450
0.400
0.350
0.300
k-ratio
10
11
12
13
14
15
16
17
18
19
20
accelerating voltage (kV)
Bastin (1986)
Bastin (1990)
P & P
Packwood
ZAF

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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