

SEMI AUX027-0213
RESULTS OF ROUND ROBIN FOR SEMI PV10, TEST METHOD FOR
INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INNA) OF
SILICON

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INAA Round Robin Report

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- 4) Wacker Chemie AG
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Contents

- Background
- Test Specimens
- Round Robin Plan
- Experimental Details
- Analysis Results
- Assessment of Results
- R&R
- Comparison with PV10
- Conclusions and Summary

Background

Background(1)

- After publication of SEMI PV 10, Test Method for Instrumental Neutron Activation Analysis (INAA) of Silicon, the PV Analytical Test Methods Task Force requested to perform a round robin for INAA based on SEMI PV10
- The following companies and laboratories kindly agreed to participate at and to support this round robin:
 - Elkem Solar AS
 - Missouri University Research Reactor (MURR)
 - SemiSol Analytik GmbH
 - Wacker Chemie AG

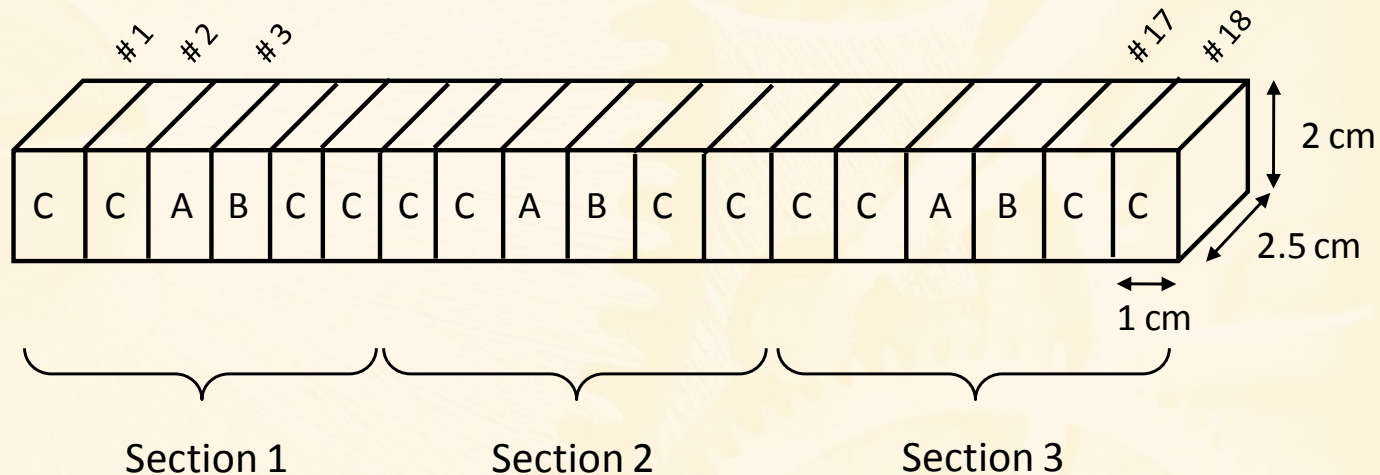
Background(2)

- Time frame of the round robin activities
 - Start: January 2011
 - Development of round robin plan: January - April 2011
 - Samples: June 2011
 - Results: November 2011 - February 2012
 - Short summary of results: March 2012
 - Final report: August 2012
- Delays occurred to due reactor maintenance and shut downs
- The contributing labs are denoted by A, B and C in this report

Test Specimens

Test Specimens

- Test specimens were kindly provided by Elkem Solar AS
- Shape of specimens: blocks with a volume of about 5 cm³ cut from a bar of purified metallurgical grade silicon



Round Robin Plan

Round Robin Plan(1)

- Laboratories A and B each got one block from each section of the Si bar, laboratory C got four blocks from each section of the Si bar, as outlined in the figure on slide 7.
- In total labs A and B got three blocks each forming three specimens, and lab C got 12 blocks forming three specimens.
- The specimens were adjusted to the specific reactor conditions, cleaned and etched as required.
- Specimens from different sections were separately irradiated at the reactors chosen by the labs and measured three times (labs A and B) or two times (lab C).
- Therefore results for the three sections contain information about the spatial distribution of impurities as well as of the repeatability of the measurements.
- In the following the specimens are identified by their section number and the lab.

Round Robin Plan(2)

- The specimens were analyzed regarding the following elements:
 - Na, K, Ca, Ti, Cr, Fe, Ni, Cu, Zn, Ga, Ge, As, Mo, Sb, W
 - Na is generated by a reaction of ^{28}Si with fast neutrons. Therefore an apparent Na concentration may be measured that is higher than originally present in the specimen. This effect depends on the reactor's neutron spectrum (SEMI PV10).
 - Ti cannot be activated by thermal neutrons, but only by fast neutrons. Its LOD (limit of detection) again strongly depends on the neutron spectrum of the reactor used for irradiation.
 - Ni is activated by thermal as well as by fast neutrons, but the nucleus activated by thermal neutrons (^{65}Ni) has a short lifetime comparable to ^{31}Si and cannot be used for analyses of Si.

Round Robin Plan(3)

- High concentrations of P, As and Sb may impact the LOD depending on the total neutron irradiation flux.
- P enhances the background in the γ -spectrum due to β -radiation.
- As and Sb also enhance the background mainly in the energy range below their γ -lines.
- The following concentrations should not be exceeded for best LOD (SEMI PV10):
 - $P < 1E16 \text{ at/cm}^3$ (2.2E5 pptw), $As < 1E13 \text{ at/cm}^3$ (534 pptw), $Sb < 1E13 \text{ at/cm}^3$ (844 pptw)
 - Other impurities in high concentration, e.g. Ge that has multiple γ -lines, also may impact the LOD for other elements.

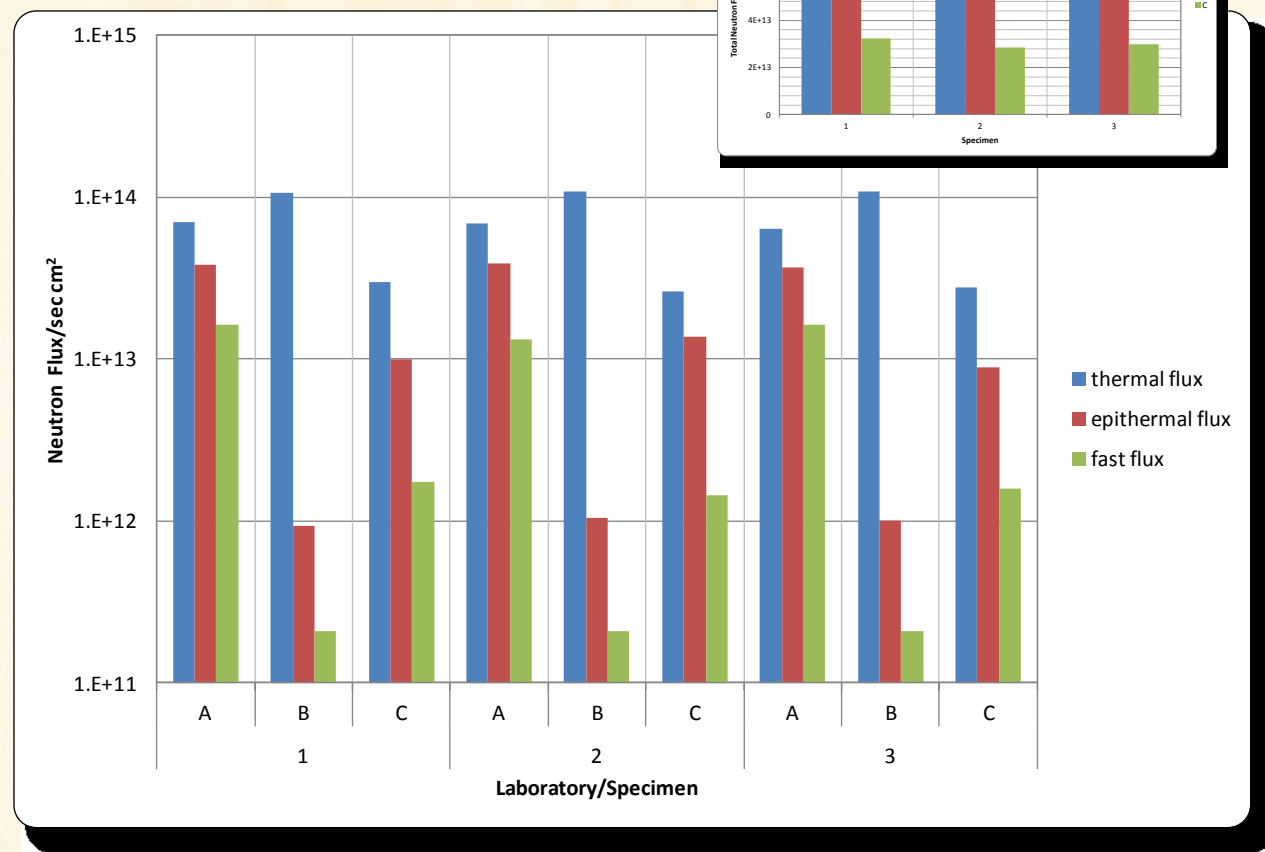
Experimental Details

Experimental Details

Lab	A	B	C	A	B	C	A	B	C
Sample	Section 1			Section 2			Section 3		
Sample mass/g	6.442	3.55	46.701	5.961	5.39	44.64	6.496	6.64	46.672
Thermal Flux/n/cm ² sec	6.95E+13	1.07E+14	2.99E+13	6.90E+13	1.09E+14	2.62E+13	6.41E+13	1.08E+14	2.76E+13
Epithermal Flux/n/cm ² sec	2.55E+12	6.20E+10	6.66E+11	2.58E+12	7.00E+10	6.09E+11	2.43E+12	6.70E+10	5.94E+11
Fast Flux/n/cm ² sec	1.62E+13	2.10E+11	1.74E+12	1.33E+13	2.10E+11	1.45E+12	1.62E+13	2.10E+11	1.59E+12
Irradiation Time/h	77.00	24.00	54.05	45.00	24.00	54.00	45.00	24.00	53.95
Waiting Time/d									
1	3.67	2.23	3.40	2.54	3.31	2.53	2.08	3.07	2.39
2	9.8	5.06	16.63	5.23	6.10	17.43	5.23	5.98	16.41
3	34.74	26.23		25.19	25.38		28.31	22.22	
Measurement Time/sec									
1	12700	7200	3600	14400	7200	3600	14400	14400	3600
2	60000	14400	43200	55000	21600	43200	29000	21600	43200
3	180000	80000		95000	80000		88500	80000	

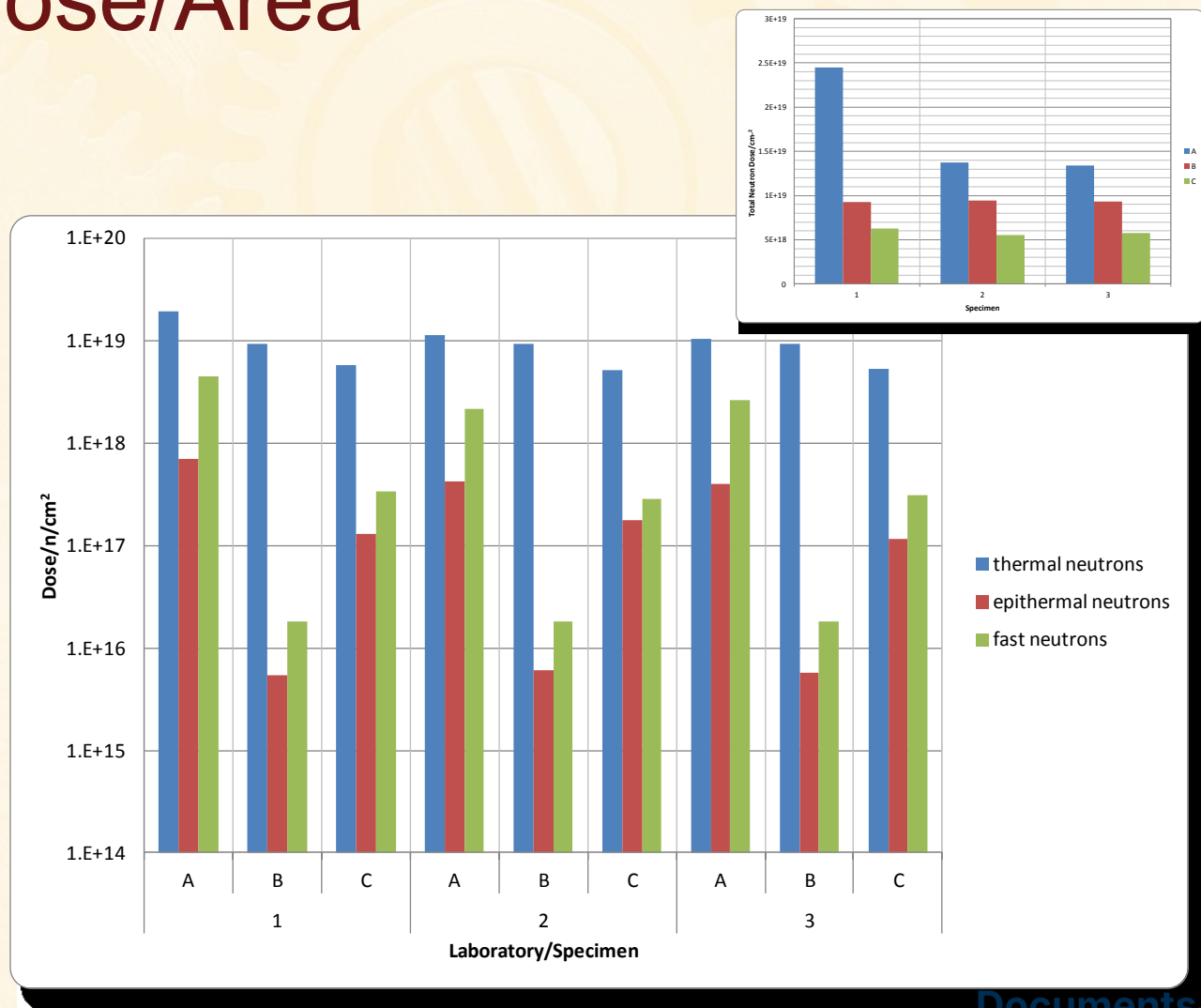
Neutron Flux

- Thermal, epithermal and fast neutron flux of the reactors used by the labs.
- Significant differences for epithermal and fast fluxes.
- The reactor used by lab A had the highest flux of epithermal and fast neutrons.
- The reactor used by lab B had the highest flux of thermal neutrons and by far the lowest flux of epithermal and fast neutrons.
- The small insert displays the total neutron flux per lab and specimen.



Neutron Dose/Area

- Neutron dose (flux times irradiation time) per area for the different reactors.
- The small insert displays the total neutron dose/cm².
- Lab A used the overall highest dose, lab C the lowest dose.
- Doses for thermal neutrons for labs A, B and C are within one order of magnitude.
- Doses for epithermal and fast neutrons differ by about two orders of magnitude between labs A and B.



Product of Specimen Mass and Flux

- The product of neutron flux and specimen mass is displayed as a measure for the activation of the short lived nuclei.
- Lab C is now at the same level as lab A for epithermal and fast neutrons due to the higher mass of the specimen measured by lab C.
- Lab C also has the largest product of mass times flux for thermal neutrons.

