INAA of 3d-metals in feedstock silicon

F. Koenn[1], U.W. Scherer[1], N. Wiehl[2], G. Hampel[2], S. Reber[3], J. Hampel[3], F. Boldt[3]
[1]Aachen University of Applied Sciences, Campus Jülich, D-52428 Jülich, Germany;
[2]Institut für Kernchemie, Johannes Gutenberg-Universität, D-55128 Mainz, Germany;
[3]Fraunhofer Institute for Solar Energy Systems (ISE), D-79110 Freiburg, Germany

Introduction: Metallurgical grade silicon (MG-Si), which is used as feedstock material for photovoltaic applications, was characterised in terms of 3d-metal impurity via INAA at the research reactor TRIGA in Mainz.

Experimental: Samples of feedstock silicon of different grades of purity and origin were provided by our collaborative partner ISE (Institute for Solar Energy Systems) in Freiburg, Germany. The purity of the provided material ranged from 99.99% (4N) till 99.999% (5N). Samples which were cleaned at the ISE by an etching solution containing nitric acid, hydrochloric acid and acetic acid, the sample material was irradiated with neutrons according two different irradiation programs and were measured by gamma-ray spectrometry.

For the measurement of manganese impurities, samples were irradiated for 30 min in the rotary specimen rack with a neutron flux of 0.17×10^{12} n s^{-1} cm^{-2}. As detection reaction ^{55}Mn(n,γ) is used, with Mn-56 as activation product, having a half life of 2.58 h. A sample changer provided additional operational capacity for the Mn measurement.

For the determination of Cr, Co, Fe and Ni, a longtime irradiation was performed. According to the half life of their activation products, the irradiation time was set to 6 h. Except for nickel, where a (n,p)-reaction was used, the above mentioned elements are activated via a (n,γ)-reaction. For irradiation at the maximal possible neutron flux at the TRIGA Mainz, the central experimental tube was chosen.

Results:
The sample material and its purity is abbreviated as follows:

- FA: Si flakes, old grade; 99.99%
- FN: Si flakes, new grade; 99.99%
- R: Si from company RIMA; 99.99%
- B: Si from company “becancour”; 99.999%

All samples were tested for their manganese impurity level, but only for FA, FN and R manganese could be satisfyingly quantified. Measured concentrations are presented in Fig 1.

The observed low level of homogeneity for the same material could be explained by non-evenly distributed micro precipitates along the grain boundaries of the polycrystalline silicon[1]. Elevated levels of manganese impurities in samples from RIMA may find their origin in the missing surface cleaning. Manganese impurity for becancour silicon could not be quantified with the method used; a detection limit of 1.6 ppbw could be determined.

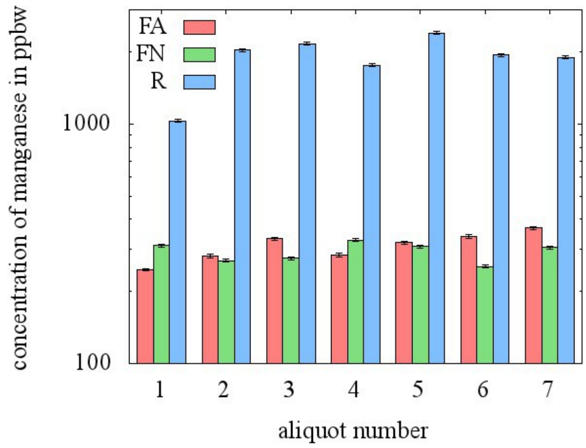

Fig 1: *Impurity levels of Mn in different aliquots of FA, FN and R (see text)*

Concentrations of Co, Cr, Fe and Ni are shown in Fig 2. Like for Mn, there is also an inhomogeneity present for the same sample material. Fe, followed by Ni, shows highest concentration in both materials, FA and FN.

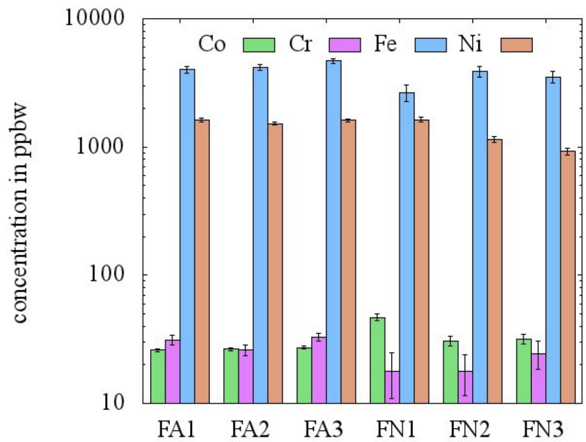

Fig 2: *Impurity levels of Co, Cr, Fe and Ni in different aliquots of FA and FN (see text)*

INAA has proven to be a valuable tool for determining 3d-metals in feedstock silicon. As a consequence of the observed inhomogeneity, more data are necessary to make a clear statement about the average concentration of the observed feedstock silicon.

References:

[1] T. Buonassisi, A. A. Istratov, M. D. Pickett, M. A. Marcus, T. F. Ciszek, and E. R. Weber: Applied Physics Letters, 89:042102, 2006.