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Detection limits for glow discharge mass spectrometry (GDMS) analyses of impurities in solar cell silicon



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ABSTRACT

The measurement of both doping elements and trace elements in solar cell silicon plays a key role for achieving high conversion efficiency of the solar cell device. Doping element concentrations in the range of few hundreds part per billions (ppb) and trace elements in the ppb or sub-ppb concentration range are typically present in multicrystalline silicon wafers for solar cells. Accurate and reliable measurements of these small amounts are not straightforward. The present work describes a fast-flow direct-current high resolution glow discharge mass spectrometer (GDMS). Detection limits for a number of impurities (B, Al, P, Ca, Ti, V, Cr, Mn, Fe, Ni, Co, Cu, Mo, Sn, W and Pb) of interest for solar cell applications have been investigated by GDMS. These detection limits are approximately 1 ppba or below, except for B, Al, P, Ca and Pb. All concentrations reported are quantitative since calculated relative sensitivity factors (RSF's) for Si matrix have been used. The detection limits have been achieved with minimum sample preparation and short analysis time.

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1. Introduction

Impurities in trace amounts degrade the properties of silicon for solar cell applications. They are mainly introduced by the feedstock material, crucible, coating, furnace components and atmosphere. Transition elements are typically present in tens to thousand of ppba in the silicon feedstock. Multicrystalline (mc) silicon solar cells are made from directional solidified ingots and, due to segregation, the level of most of the impurities in the final mc-Si wafers (prior to solar cell processing) is less than tens of ppba. Dissolved transition elements introduce deep levels in the band gap thus increasing the carrier recombination rate. For some common impurity elements, e.g., iron, chromium and titanium, this effect is significant at concentrations on the order of a few ppba [1–6]. Davis et al. [1] studied several impurities in single-crystalline (sc) silicon made by the Czochralski process and reported on their effect on solar cell efficiency. According to their investiga-

tion, the addition of 1 ppba Ti or 140 ppba Fe or 100 ppba Cr to the silicon wafers causes 30% decrease of the normalized solar cell efficiency. Istratov et al. [7] and Macdonald et al. [8] reported on the typical levels of impurities in mc-Si wafers and showed that the levels of Fe, Cr, Cu, Ni and Co range between 3×10^{12} to $3 \times 10^{14} \text{ cm}^{-3}$ (approximately 0.1–10 ppba). Coletti [9] reported on the effect of Fe, Cr, Ni and Ti on multicrystalline silicon solar cells. Recently, the level of impurities in the so-called solar-grade silicon (SoG-Si) feedstock has been the focus of many investigations [4,10,11]. It was shown [11] that a SoG-Si feedstock containing 83 ppba Al and 99 ppba Ti will give a 3% relative reduction of solar cell efficiency. Specifications for SoG-Si feedstock are currently under discussion. The main attention is given to the concentration of doping elements, e.g. B and P, since they are more difficult to remove during directional solidification and since they directly affect the solar cell base resistivity. Typical levels of B and P in undoped SoG feedstock materials are <130 ppba and <90 ppba, respectively [9].

Being able to measure, hence control, the concentration of both the doping elements and trace elements in solar

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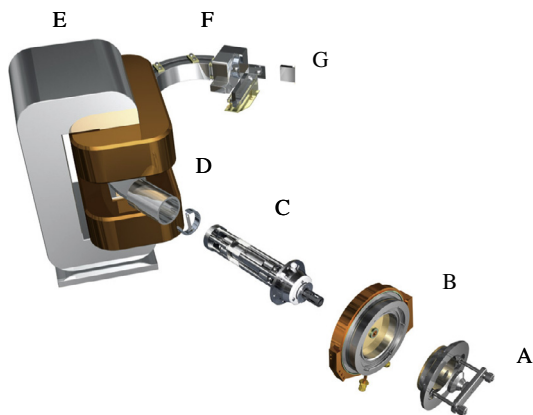


Fig. 1. GDMS main components: A – sample holder; B – plasma chamber; C – ion optic assembly; D – resolution slit; E – magnet; F – electro-static analyser; G – detector. (Courtesy Thermo Fisher).

cell silicon play a key role for achieving the highest energy conversion efficiency of the devices. However, as indicated above, these amounts are quite low. Therefore, accurate and reliable measurements of these materials are not straightforward. The techniques to analyze transition elements in the ppba level in silicon include neutron activation analysis (NAA), micro X-ray fluorescence spectroscopy (μ -XRF), glow discharge mass spectrometry (GDMS), secondary ion mass spectrometry (SIMS) and, to a certain extent, also inductively coupled plasma mass spectrometry ICP-MS. However, in terms of sensitivity and quantification capabilities as well as easy to perform the analysis in a relatively short time, the GDMS instruments are superior to analyse conducting and semiconducting samples in the low ppb range compared to the other techniques. Currently, the GDMS instruments are receiving increasing attention for the analyses of materials for photovoltaic (PV) applications [12,13]. However, no data have been reported for the limits of the detection and capability of these instruments. Therefore, in the present work the determination of detection limits of GDMS analyses for several elements of interest in PV silicon are described and discussed in details.

2. Experimental

2.1. The GDMS instrument

The instrument used in this study is a fast-flow direct-current high resolution glow discharge mass spectrometer (GDMS). Fig. 1 shows the main components of the GDMS [14,15]. In this type of instruments, the sample forms a cathode in a low pressure gas discharge, Argon in this study. The glow discharge ion source is a Grimm-type, which allows for both surface and in-depth analysis by controlling the discharge parameters to obtain planar sputtering [16]. The samples must have a flat and smooth surface, and are loaded in the sample holder. Ions generated in the plasma chamber are focussed by the ion optic assembly onto the resolution slit before entering the magnet. After

the mass separation by the ions' momentum in the magnetic field, the electro-static analyser (ESA) serves for an energy separation to achieve high resolution capabilities required for separation of polyatomic interference species from the analyte signals. Finally the ions on their respective masses are counted by the detection system. Different kinds of ionization processes occur in the plasma chamber as indicated in Fig. 2. The Element GD has three detector modes, namely the counting and analog detector obtained from a secondary electron multiplier (SEM), and the Faraday detector. The resolution slit allows choosing between low ($R = 400$), medium ($R = 4000$) and high ($R = 10,000$) resolutions (i.e. $m/\Delta m$). Due to the ratio of the mass of each element over its charge, m/Z , the ions are separated by the analyzer.

2.2. Quantification

The intensity of the element signal relative to the matrix signal allows quantification of the concentration of each element. When trace elemental analyses are performed on high purity materials, such as in this study, the ion signal for the matrix (I_M) is assumed to be large relative to the individual elements (I_X). Therefore, the matrix ion current is a good approximation of the total ion current, and the matrix can be assumed to have concentration of 100%. The concentration K of an element X , K_X , can be calculated as [17]:

$$K_X = \left(\frac{I_X}{I_M} \right) K_M \left(\frac{A_M}{A_X} \right) \quad (1)$$

where K_X and K_M are the atomic mass fraction of the element and matrix, respectively; I_X and I_M are the ion beam signals of the element and matrix, respectively; A_X and A_M are the isotope abundances of the element and matrix, respectively.

In order to obtain quantitative results, a concentration dependence of element-specific and matrix-specific sensitivity factors (named relative sensitivity factors, RSF's) has to be known. The element concentration in mass unit, C_X , can be expressed as:

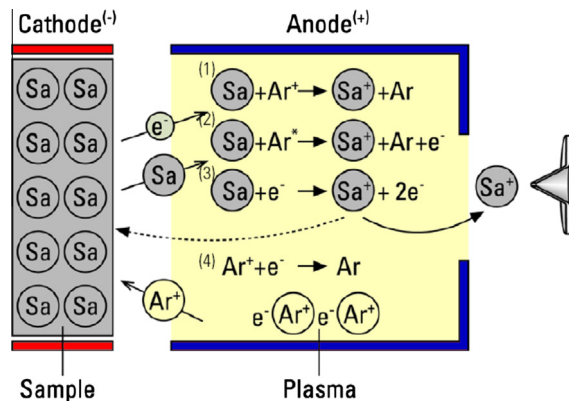


Fig. 2. Schematic view of the plasma chamber with the sample (cathode) on the left and the cone on the right. (1) to (4) are the ionization processes occurring in the chamber (Courtesy Thermo Fisher).

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