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Development of boron calibration via hybrid comparator method in prompt gamma activation analysis

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Abstract

The prompt gamma activation analysis (PGAA) facility at the Nuclear Engineering Teaching Laboratory at The University of Texas at Austin was utilized to quantify boron concentrations in boron carbide semiconductor films deposited on silicon substrates. Calibration was complicated by the unique and varying sample geometries analyzed. In addition, there was a dearth of solid materials available with quantified boron concentrations having comparable or readily modifiable dimensions to exploit for calibration purposes. Therefore, a novel hybrid comparator method was developed for the quantification of boron utilizing aluminum as an inexpensive and easily machinable reference material. Aluminum samples were manufactured with high tolerances to match the geometry of each sample of interest. Each boron carbide film sample and its congruent aluminum sample were measured in the PGAA system. The measured aluminum responses and relevant nuclear parameters were used to standardize the

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measurements. A boron standard was created using a procedure derived from a similar approach used by the National Institute of Standards and Technology. Quality control measurements using this standard show that the method provided accuracy to within 5% for boron quantification.

Keywords: Prompt gamma activation analysis (PGAA), Boron, Calibration

Introduction

Prompt gamma activation analysis (PGAA) has become a powerful nuclear analytical technique that continues to demonstrate value in an increasing number of applications [1]. The fundamentals, practice, and key advancements in applied methods of PGAA are well documented in the literature [2, 3]. With reported detection limits of less than 10 ng g⁻¹ in some materials [4], PGAA is one of the most sensitive, non-destructive analytical techniques available for low-level boron quantification. This makes PGAA a very attractive option for quantitative analysis of boron in thin-film semiconductors, where deposition characteristics and boron concentration of thin-film growth are crucial to device function.

A big challenge is obtaining an accurate characterization and efficiency calibration of the system. Absolute (parametric) and relative calibration (or standardization) methods are largely considered inaccurate or impractical in most analyses, although strict parameterization approaches have been pursued [5]. The accuracy and applicability of more appealing comparator-type and k_0 -type standardization methods originating in neutron activation analysis (NAA) are well established [6–8] and consequent efforts leading to successful implementation of these calibration techniques in standard PGAA practice have been described [9, 10].

In a recent study, boron carbide thin film samples were analyzed for absolute boron concentrations using the PGAA facility at the Nuclear Engineering Teaching Laboratory (NETL) at The University of Texas at Austin. Calibration of the system was complex due to a number of factors, which include the following: large number of samples with different shapes and sizes, spatially nonuniform neutron flux profile at sample location [5, 11], absence of boron solid material standards with comparable geometries and properties (absorption and scattering), and a required quick turnaround with limited expense. Furthermore, at the onset of analysis, characterization of the silicon substrates on which the

boron carbide films were deposited was limited. It was known only that boron carbide film deposition was uniform and boron concentration per unit area of film was $\leq 250 \mu\text{g cm}^{-2}$ for each sample. In a strict sense of their definition, neither the comparator nor the k_0 calibration method was optimal in itself for these experimental constraints.

In this paper, the boron calibration method developed and utilized in this study of boron carbide films using PGAA is presented. The validity of the results using this approach are examined through an internal comparison of known silicon content in the substrates, which was acquired only after completion of the original analysis, as well as in comparison with a carefully constructed in-house boron standard. The results confirm that this method is capable of achieving accurate measurements of boron in thin-film semiconductors at minimal material expense with promising application in other elemental analyses.

Theory

In PGAA measurements, count rates from experimentally detected gamma rays of a characteristic energy are used to determine elemental concentrations of the materials analyzed. For most PGAA facilities and practical applications, the conditions in which these measurements are made permit a number of simplifications to be made in the analysis. In this study, the boron carbide thin film and silicon substrate is treated as a homogenous volume (to be later confirmed) over the small sample thickness. Neutron self-shielding in the sample due to boron is deemed negligible as less than 1% of the neutron flux is attenuated through the sample given the maximum film thickness, maximum possible boron concentration stated, and the boron total interaction cross-section. The PGAA facility neutron flux is sub-thermal with the faster neutron component removed due to the 6 m long, curved neutron guide [12]. For the light elements composing each sample matrix, gamma-ray self-absorption is negligible, especially as the samples are positioned at 45° with respect to the incident neutron beam and high-purity germanium (HPGe) detector face. Based on these assertions, for a given set of experimental conditions, the net peak area A_x from a neutron capture gamma ray of energy E_c from element x , can be expressed as a product of constant terms [3, 9]:

$$A_{x,E_\gamma} = \left[\frac{N_A m_x}{M_x} \right] \varepsilon_{E_\gamma, V} \theta_x \Gamma_{x,E_\gamma} t_l \Phi_{th} g \sigma_{o,x} \quad (1)$$

where N_A is Avogadro's number, M_x is the atomic weight, m_x is mass of element x , $\varepsilon_{E_\gamma, V}$ is the detector's efficiency within volume V of sample, θ_x is the isotopic abundance, Γ_x is the gamma-ray yield in photons per capture, t_l is the detector live time during irradiation, Φ_{th} is the thermal flux, g is the Westcott factor, and $\sigma_{o,x}$ is the thermal neutron capture cross-section.

In addition, the Westcott factor $g \approx 1$ for all isotopes of interest in this study and so behave as good $1/v$ absorbers, which removes the dependence in Eq. (1). Using the definition of the partial elemental capture cross-section $\sigma_{\gamma, E_\gamma}^x$ [3], and rewriting the bracketed terms as the number density N_x of element x since the mass density ρ_x of any element x is known, Eq. (1) can be written as:

$$A_{x,E_\gamma} = N_x \varepsilon_{E_\gamma, V} \Phi_{th} \sigma_{\gamma, E_\gamma}^x \quad (2)$$

Normalizing the net peak area of element x to a comparator element c , the following is obtained:

$$\frac{[A_{x,E_\gamma}/t_l]_x}{[A_{c,E_\gamma}/t_l]_c} = \frac{[N_x \varepsilon_{E_\gamma, V} \Phi_{th} \sigma_{\gamma, E_\gamma}^x]_x}{[N_c \varepsilon_{E_\gamma, V} \Phi_{th} \sigma_{\gamma, E_\gamma}^c]_c} \quad (3)$$

As noted, the neutron flux at the sample location is spatially non-uniform, however, as long as element x and comparator element c are homogeneously mixed in sample, then the flux cancels out of Eq. (3). This is also true if element x and c are in separate samples placed in the exact same location in front of the neutron beam and have similar neutron and gamma-ray self-shielding properties (each sample matrix has similar nuclear interaction compositions) and all of the above assumptions are accurate. This also removes the V dependence in the efficiency term ε for both elements. On a final note, the variation in flux intensity over time due to changes in the reactor operating conditions must be accounted for in every PGAA measurement as this can occur over the course of a long count and especially between day-to-day irradiations. A chief contributor to this variation is thought to be ^{135}Xe buildup in the reactor core [13]. These variations are accounted for by normalizing the

counts in each PGAA measurement to the neutron counts acquired by a proportional ^3He detector located between the curved neutron guide and focusing element [11, 13]. The neutron counts from the ^3He detector must also be normalized in each measurement to the HPGe detector real time during the irradiation before being directly compared to background or other comparator element measurements. As such, Eq. (3) can be rewritten and solved for the unknown atom density N_x of element x by comparison with an internal (or separate external sample of) comparator element c as follows:

$$N_x = N_c \frac{[(A_{x,E_\gamma} t_r) / (t_l n_x)]_x [\varepsilon_{E_\gamma} \sigma_{\gamma,E_\gamma}^c]_c}{[(A_{c,E_\gamma} t_r) / (t_l n_c)]_c [\varepsilon_{E_\gamma} \sigma_{\gamma,E_\gamma}^x]_x} \quad (4)$$

where t_r is the HPGe detector real time during the irradiation and n_x is the number of neutrons detected by the ^3He detector. Note this assumes that background contributions have already been subtracted out from the quantity represented by the terms in brackets. Equation (4) is then used to determine the unknown mass of element x in all PGAA measurements, whether the comparator element is internal to the sample containing element x or external in another sample of identical geometry.

In this study, both ^{27}Al and ^{28}Si are used as comparator elements to determine natural boron elemental concentrations in the boron carbide thin films and the results are compared. The ^{27}Al is present in known quantities as external comparator samples composed of 6061-T6 aluminum alloy, which are easily fabricated with identical geometry to each boron carbide sample. The ^{28}Si is present in the silicon substrates on which the boron carbide films are deposited and are treated as an internal comparator that is homogeneously mixed in each sample.

The boron gamma-ray peak of interest is the 477 keV photopeak emanated from the $^{10}\text{B}(n, \alpha)^7\text{Li}$ reaction, which is Doppler-broadened (460–490 keV) due to the recoiling ^7Li nucleus after alpha particle emission. This peak cannot be fit using conventional Gaussian peak-fitting software and detailed deconvolution and treatments of peak-fitting routines for the Doppler-broadened boron peak shape have been discussed in the literature [14, 15]. However, as in the study by Harrison and Landsberger in 2009 [16] involving the determination of boron over a large dynamic range using the NETL PGAA facility, the use of a simple peak-summing technique to measure boron peak area in the current study was found to introduce less than 1% error. The major interference contributors to

the 477 keV boron peak are expected to be germanium (467.6, 468.1, 468.8, 470.0, 472.1, 475.4, 476.8, 479.8, 481.7, 484.6, 488.3 keV) due to scattering of neutrons off the sample into the HPGe detector crystal, sodium (472.2 keV) from human contamination, and silicon (477.1 keV) from the substrate upon which the boron films are deposited. Using the cross-section ratio and net peak area at a separate, interference-free peak (different energy gamma ray) of the same element, the contribution of that element as an interference to the Doppler-broadened boron peak can easily be determined. Again, interferences were determined to be insignificant for all isotopes of interest and the use of a peak-summing technique for boron was found to be accurate in this study.

Experimental

All experiments were conducted using the NETL PGAA facility at The University of Texas at Austin. The PGAA facility is equipped with the Texas Cold Neutron Source (TCNS), which has the ability to pass neutrons streaming out of beam port #3 on the TRIGA Mark II research reactor through a moderating chamber, which is cooled to 27 K. However, these experiments did not require an increased sub-thermal neutron flux fraction, so the TCNS was not in operation during these experimental measurements and the moderating chamber was purged with helium instead. A 6 m long neutron guide with a 300 m radius of curvature preferentially directs low velocity neutrons toward the sample through biological shielding at a wavelength of 4.35 Å and effective temperature of 39 K [11]. The collimated thermal-equivalent neutron flux reaching the sample position has been measured previously at $5.32 \times 10^6 \text{ cm}^{-2} \text{ s}^{-1}$ when the reactor operates at a power of 950 kW [11]. The resulting gammas from the irradiated sample are measured by a horizontal, p-type, 65% efficient ORTEC GMX Series High Purity Germanium (HPGe) detector. Detailed Schematics and characterization of the facility including background contributions affecting detection limits are published elsewhere [11, 13].

While the facility is equipped with a number of different sample holding devices [17] the wide variation in size and geometry of samples required for the boron carbide semiconductor studies rendered precise and reproducible placement in the spatially non-uniform beam

impossible. Therefore, custom sample holders were created to allow samples of varying dimensions and their geometrically analogous comparator to experience an identical incident neutron flux. The holders were fabricated out of 6061-T6 aluminum alloy with a threaded rod with screw and nut assembly body allowing sample height and angle to be adjusted as well as locked into place. Four locating pins on the base further stabilize the sample and ensure consistent placement into a fixed baseplate. Interchangeable head plates were fabricated in order to accommodate both rectangular and circular boron carbide semiconductor geometries. The head plate machined for circular geometries was designed with a concave internal surface to match the radius of curvature of circular disk samples, while a locating pin was utilized in the head plate for rectangular samples.

Comparator samples for each unique geometry were constructed out of easily machinable aluminum alloy, which is also a more-realistic and currently studied substrate material for possible large-scale production of $^{10}\text{B}_4\text{C}$ thin films used in gaseous neutron detectors [18]. The cost and material properties of aluminum allowed for inexpensive, timely construction of a large number of comparators using standard machine shop tools.

In the case of the in-house standard, a known mass of certified aqueous boron standard material (Inorganic Ventures, MSB-100PPM) was deposited onto filter paper affixed to an aluminum substrate sample, which ensured that all liquid remained on the surface and that the boron was uniformly deposited across the surface area of the sample. Excess liquid solvent was then evaporated in a vacuum oven resulting in a thin, uniformly distributed boron layer atop the aluminum substrate. This process of deposition and solvent evaporation on an absorbing material substrate is derived in part from steps used by the National Institute of Standards and Technology (NIST) to create solid boron standards in pellet form [4].

The characteristic gamma ray of interest in the PGAA measurements was ^{27}Al (1779 keV) for the 6061-T6 aluminum alloy comparators and ^{28}Si (3539 keV) for the silicon substrate internal comparators. It is important to note that in this study the ^{27}Al (1779 keV) gamma ray is not prompt; it has a 2.24 min decay. In order to account for this non-prompt gamma-ray emission, counting is delayed after opening the neutron beam shutter by a minimum of 30 min.

Procedure

Prior to the acquisition of PGAA sample spectra, the detector efficiency was calculated using a similar method as described in Biegalski et al. [5]. A NIST traceable ^{152}Eu point source was utilized to calculate a base efficiency curve for the sample. An iron foil was then measured in the PGAA facility and a relative efficiency curve was calculated out to 7646 keV. The ^{152}Eu and the Fe foil based efficiency curves were normalized and merged. The final efficiency fit was calculated utilizing a least-squares fitting algorithm to create a fourth-order polynomial in lognormal energy and count space. The relative uncertainty in the efficiency curve is estimated to be $\pm 1.2\%$ based on propagation of uncertainty from the peak counting statistics, source activity uncertainty, gamma-ray yield uncertainty, and the χ^2 value for the fit.

Depending upon the general shape of the sample, either the disk or square head plate was attached to the sample holder base plate and a background spectrum was acquired. In order to align the sample within the incident neutron beam, a mock-up replica of the general sample geometry was created using a Formlabs stereolithography (SLA) 3D printer, which features either a peripheral slot around the sample perimeter for inserting a cadmium wire or a circular inset for attaching a cadmium disk on the sample surface. Using this replicate with cadmium insert, images were captured by a neutron radiography camera placed directly behind the sample holder with the beam port shutter open as shown in **Fig. 1**. Adjustments were made to the sample holder until the sample was correctly aligned.

After aligning the neutron beam with a particular sample geometry, the actual boron carbide sample was placed in the sample holder and a spectrum was acquired, immediately followed by the irradiation of its corresponding aluminum comparator.

Results and discussion

A total of ten boron carbide thin-film samples, which had been deposited on silicon substrate backings, were analyzed. Another ten comparator samples composed of 6061-T6 aluminum alloy, each having identical dimensions to a boron carbide sample, were also analyzed in order to standardize the PGAA measurements for boron content. The surface



Fig. 1. Process of aligning each sample within neutron beam using 3D printed replicates of sample geometries with cadmium inserts and a neutron radiography camera

area of the boron carbide films ranged between 2.8 and 20.2 cm² with a total sample mass between 0.4 and 1.82 g (including silicon substrate). Total sample thickness varied slightly in the sample set between 0.036 and 0.038 cm, which also includes the silicon substrate. Using the net

Table 1. Determined boron mass in each sample via aluminum as an external comparator element and silicon as an internal comparator element in PGAA measurements.

Sample number	Boron mass (μg) determined by		% Difference
	External comparator (^{27}Al)	Internal comparator (^{28}Si)	
1	2886 ± 133	2847 ± 134	1.4
2	2270 ± 105	2270 ± 107	0
3	302 ± 14	298 ± 14	1.3
4	1192 ± 55	1245 ± 59	4.4
5	56 ± 3	56 ± 3	0
6	36 ± 2	38 ± 2	5.6
7	28 ± 2	29 ± 1	3.6
8	36 ± 2	38 ± 2	5.6
9	48 ± 2	48 ± 2	0
10	3.1 ± 0.3	3.2 ± 0.3	3.2

counts of the ^{27}Al (1779 keV) characteristic gamma ray from the comparator sample spectrum and net counts of the ^{10}B (477 keV) and ^{28}Si (3539 keV) characteristic gamma rays from the boron carbide sample spectrum, the total boron mass was determined via two different reference elements for each sample. The results using aluminum as an external comparator and silicon as an internal comparator are presented and compared in **Table 1**.

A comparison of the total boron mass determined from the external aluminum comparator sample versus the internal silicon substrate content for each thin film analyzed in Table 1 clearly demonstrates a strong agreement between both approaches over a notable range of boron mass deposition. This supports initial assumptions that neutron and gamma-ray self-shielding effects within the sample are negligible in PGAA measurements of boron carbide thin films of similar characteristics to within the desired uncertainty. As further validation of this hybrid comparator method, the detector responses for the external aluminum comparator samples were also utilized in the same approach to calculate the mass of silicon substrate in each boron carbide sample. These results are compared with the known amount of silicon in each substrate in **Fig. 2**, which was disclosed shortly after the original PGAA experiments by the research team responsible for creating the boron carbide thin-film samples in the Department of Electrical and Computer Engineering at the University of Nebraska-Lincoln.

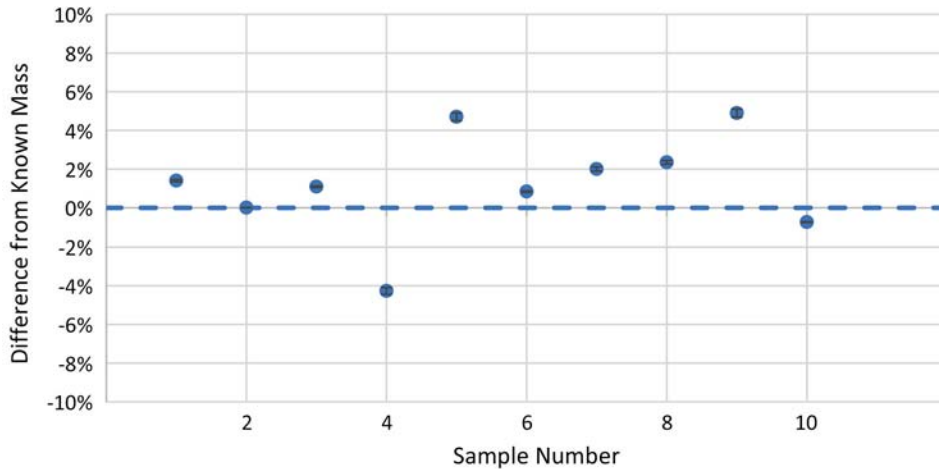


Fig. 2. Comparison of known silicon content in each silicon substrate with PGAA measurements using aluminum as the reference comparator material

Again, the results in Fig. 2 provide further confirmation of the boron mass results obtained using either the external aluminum comparator samples or internal silicon substrate content to standardize the PGAA boron measurements. As a final, more-direct effort to validate boron concentrations obtained using this method, an in-house standard with precisely measured boron content was created and analyzed using the same PGAA procedure with an aluminum external comparator sample having the same dimensions. The calculated boron mass (25.98 ± 1.30) was within 2.1% of the known boron mass (25.45 ± 0.19), which demonstrates a sufficient level of accuracy in the measured boron content using this efficiency calibration method for the PGAA system.

Conclusions

A hybrid comparator method for boron calibration in PGAA measurements of boron carbide thin films deposited on silicon substrates has been introduced and benchmarked using two different implementations of a reference comparator material. The results show that accurate measurements of boron content in thin films are achievable to within approximately 5% accuracy of total boron mass. Also shown is the justification of initial assumptions made in simplifying the PGAA measurements using this method of analysis for boron carbide thin films of equivalent

characteristics including negligible neutron and gamma-ray self-shielding effects, homogenous material matrix, and removal of spatially non-uniform flux dependence on calculations through the use of precise and repeatable sample placement within the incident neutron beam. This calibration method offers the capability of achieving accurate PGAA measurement of boron in thin films without the need to create an expensive reference standard for each sample of different geometry and may significantly reduce the turnaround time for completing the analysis of a large sample set. Significant promise in the application of this method for evaluating other thin-sample material compositions has also been demonstrated.

Although the accuracy of PGAA measurements in this study has only been confirmed to within approximately 5% of the calculated boron mass, it is believed that this method is capable of even higher levels of precision for boron as well as other elements. Future efforts are intended to reduce the uncertainties in the aforementioned measurements by reducing contributions from human error in sample placement and repeatability, inhomogeneity of boron in the thin film growths, uncertainty in concentration of comparator element in samples, and further addressing nonlinearities in the ratio of detector response to element mass due to the smaller influences in neutron and gamma ray self-shielding effects in thin-film samples. Other efforts will be directed upon the possibility of expanding this hybrid comparator approach to other thin-film elemental compositions.

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