

Quantitative analysis of the metallic contamination on GaAs and InP wafers by TXRF and ICPMS techniques.

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Due to their specific optical properties and their high electron mobility, GaAs and InP semiconductor crystals are used in various applications: in optoelectronics field (LED, laser diodes), in high power and high frequency electronics and also for high efficiency solar cells.

The surface cleanliness of such semiconductor is a major requirement for processes control (e.g. surface preparation and cleaning, high quality epitaxial films) and then devices performances. As a result the metallic contamination of these III/V substrates and its control appears more and more as a significant concern. Unlike to Si wafers for which in-line TXRF (Total reflection X-Ray Fluorescence) as well as chemical collection by LPD (Liquid Phase Decomposition) or HF VPD-DC (Vapor Phase Decomposition-Droplet Collection) combined with ICPMS (Inductively Coupled Plasma Mass Spectrometry) were largely used and well documented, only TXRF is mentioned to measure metallic contamination on InP or GaAs in literature without detailed report [1,2]. In this paper, we investigate the ability to characterize metallic contamination on GaAs and InP wafers both by TXRF and ICPMS techniques.

To develop such analyses, prime 100mm wafers of GaAs 6°B (supplied by AXT) and InP 2°B doped S (supplied by Sumitomo) were used. Both clean wafers (as-delivered wafers) and controlled contaminated wafers by spin coating method in the range  $10^{11}$ - $10^{13}$  at.cm<sup>-2</sup> were used.

For chemical collection, home-made LPD system and VPD reactor were used. Droplet collection was carried out manually. A diluted HNO<sub>3</sub> solution was selected as collection solution both for LPD and VPD. Analysis was performed with an Agilent ICPMS7500cs. The Collection Efficiency (CE) of metals was determined by consecutive LPD-ICPMS or VPD-DC-ICPMS and was also confirmed by pre and post TXRF analysis.

TXRF analysis was evaluated from a Rigaku TXRF300A using a W rotating anode enabling analysis from Na to U with different X-ray excitation beam (beam 1 : W M $\alpha$  @ 1.77keV for Na, Mg and Al; beam 2: W L $\beta$  @ 9.67keV for P to Zn notably; beam 3 : W high energy @ 24keV for heavy metals e.g. Zr, Mo, Ta, W, Au, Pt...). When TXRF analysis is possible, analytical conditions (in terms of incident angle, azimuth angle...) were then optimized to minimize background signal and reach the better sensitivity. TXRF calibration was carried out from controlled contaminated wafers with Al, Ni and Mo (respectively for Beam 1, 2 and 3) and in reference to ICPMS quantification.

In case of GaAs, both VPD-DC-ICPMS and TXRF analyses were implemented. Regarding the first one, the VPD-DC step can be performed thanks to a sufficient GaAs hydrophobic surface for droplet collection. Then, we demonstrated that VPD-DC-ICPMS allows well the characterization of usual contaminants. Indeed, high collection efficiencies (>90%) was obtained except for Cu and therefore noble metals (see fig 1), and very relevant detection limits for contamination control were obtained between  $10^8$ - $10^{10}$  at/cm<sup>2</sup> depending on element.

TXRF analysis on GaAs is limited by L and K X-Ray emission lines of As and Ga. Beam 1 is fully unusable and

Beam 3 spectrum is interfered in the analysis range of Au, Pt and Ge. As a result, Na, Mg, Al, Ge are not analyzable and Au and Pt analysis must be performed on secondary X-Ray lines, leading to high detection limits (few  $10^{12}$  at/cm<sup>2</sup>). Calibration was performed successfully and K, Ca and usual transition metals including Cu can be analyzed with detection limits in the range of few  $10^{10}$  -  $10^{11}$  at/cm<sup>2</sup>.

For InP wafers, the droplet collection can't be performed due to the hydrophobia lack of the surface after HF VPD. Thus, a LPD step was then implemented despite the larger solution volume (few ml) and the sensitivity loss induced. The LPD-ICPMS method implemented leads to good CEs (>85%) for usual metallic contaminants except Cu allowing their characterization with interesting LDs ( $10^9$ - $10^{11}$  at/cm<sup>2</sup> range).

TXRF analysis on InP is possible for most of elements except for K, Ca because these ones are interfered by L In lines fluorescence. Then, calibration was carried out successfully (cf. fig 2) However, despite analytical conditions optimization, sensitivity is moderately interesting with detection limits in the range of few  $10^{11}$ - $10^{12}$  at/cm<sup>2</sup> except for Cu, Ni and Zn with LDs  $\sim 6.10^{10}$  at/cm<sup>2</sup>.

Finally, TXRF and ICPMS methods were implemented to characterize metals traces on GaAs and InP. Analyzable elements and performances of each method were determined, showing that the combination of both techniques enable to cover main usual contaminants (Al, Na, Ca, Ti, Cr, Fe, Zn, Cu...) as well elements as Pt, Au. Improvement perspectives will be then discussed.

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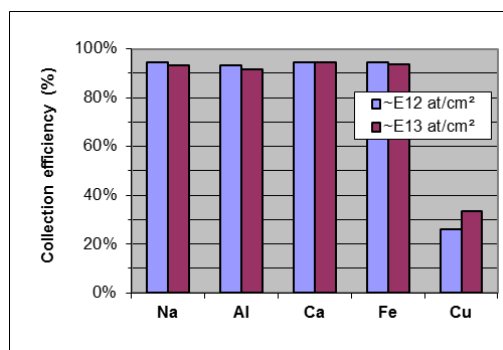


Fig. 1: Collection efficiencies of VPD-DC step on GaAs for some metals at the levels of  $10^{12}$  and  $10^{13}$  at/cm<sup>2</sup> range

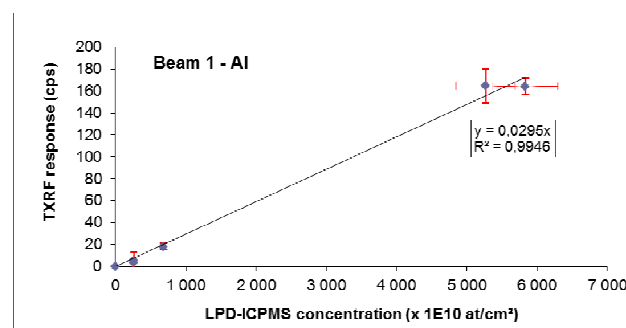


Fig. 2: TXRF calibration on InP for Al (beam 1) from contaminated wafers in reference to LPD-ICPMS.