A Novel Approach to Silicon Carbide (SiC) Material Characterization

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As an advanced ceramic material, silicon carbide (SiC) has become a promising and attractive substrate to the semiconductor industry for making high-performance electronic devices. Because of its high electric and mechanical strength, thermal and chemical stability, and radiation resistance, SiC based devices are capable of operating under extreme conditions at high temperature, under radiation conditions, or in chemically active media. Additionally, metal semiconductor field effect transistors (MESFETs) fabricated on high-resistivity SiC substrate have shown a record of performance while biased at a high operating voltage [1]. However, the integrity of the SiC depends highly on the trace residue impurities in the materials. Analysis of SiC materials and coatings for trace impurities has therefore become a prerequisite to quality control of raw materials, crystal growing environment, and coating deposition processes.

The analysis of SiC materials has traditionally been done by ICP-OES and ICP-MS due to their excellent accuracy and precision as well as the use of NIST traceable elemental standards for instrument calibration. If there is a shortcoming in their analysis, it is the general requirement that SiC material be in solution form prior to the analysis. However, SiC is a refractory ceramic which is very difficult to be dissolved. Even with a mixture of HNO_3, HF, and fuming H_2SO_4 at high pressure, the chemical dissolution process can take days to a week. The sensitivity of this method for trace impurities is relatively low due to process contamination (high blanks) and a large dilution required prior to the analysis. Fusion has been reported as a quicker method for the decomposition of SiC. However the use of alkaline oxidative mixtures of NaOH or Na_2CO_3 with KNO_3 and Na_2O_2 prevents laboratories from analyzing a large number of key elements in the SiC material accurately because of the process contamination, impurities present in the reagents and analyte loss arising from the fusion process. The analysis accuracy of the fusion method is also compromised due to an incomplete acidic re-dissolution. Therefore, the analysis of SiC materials has remained very challenging and the current available methods are still very problematic.

At Air Liquide – Balazs Nanaonaalysis, we have been developing new direct solid analysis methods in order to overcome the process contamination and dissolution problems in analysis of SiC materials and coupons. The specific techniques that were studied included glow discharge optical emission spectroscopy (RF GD-OES) and laser ablation ICP mass spectrometry (LA ICP-MS) [2]. These techniques enable us to directly analyze the solid SiC materials and coupons in their natural states without going through the wet dissolution process. By utilizing RF plasma or laser ablation for material sputtering, excitation or ionization, many intrinsic limitations associated with chemical dissolution are avoided.

For example, contamination from chemical reagents and processes is minimized; sample dilution prior to analysis and analyte loss are avoided; the detection limits for trace elements in SiC is greatly improved; and local analysis and depth profiling of SiC materials is facilitated. As a result of our research and developments, we can not only quantitatively analyze any SiC material for more than 70 trace elements at ppb (ng/g) - sub ppm (µg/g) levels but also spatially investigate elemental distributions for major elements, impurities and dopants in three dimensions.

RF GD-OES, with its nm depth resolution and simultaneous multi-element profiling capability, is used to profile surface, near surface and interface (e.g. oxide and SiC interface) of SiC materials. The depth profiles obtained have been successfully used to assist with ion implantation processes and doping control in SiC. LA ICP-MS is used for microscopic defect identification and quantitative determination of trace residue impurities in bulk SiC. The information obtained has been used to help QC raw material, SiC crystal purification, and EPI layer preparation. The signal intensities produced by both techniques all have simple and well-defined mathematical (linear) relationships with elemental concentrations in the material. Each technique has a wide linear dynamic range and when coupled with the various NIST traceable material standards that our laboratory has developed, accurate and precise surface and bulk SiC analyses are possible.

References:

1. G Augustine, V, Balakrishna, and C.D. Brandt, Journal of Crystal Growth, 211, 2339, 2000.
2. F. Li and S. Anderson, Frontiers of Characterization and Metrology for Nanoelectronics, D.G. Seiler et al Ed., American Institute of Physics, 62-66, 2009