

General ToF-SIMS experimental description. Please choose the right experimental setting used in your study (HR spectra, HR images or depth profiles). Please check with me Cs sputtering energy, current and mass calibration if you are not certain on the values.

ToF-SIMS¹ is a highly sensitive surface analytical technique for acquisition of elemental and molecular information from the surface of a material with high spatial and mass resolution. A finely focused, pulsed primary ion beam is rastered across the surface of the sample and the secondary ions emitted at each pixel are extracted into a time of flight mass spectrometer, mass filtered, and counted. With dual beam operation, depth profile of ions can be obtained and quantification of elements can be achieved with standard. (This can be put in introduction if ToF-SIMS has been extensively used in your study).

ToF-SIMS analyses were conducted using a TOF SIMS V (ION TOF, Inc. Chestnut Ridge, NY) instrument equipped with a Bi_n^{m+} ($n = 1 - 5$, $m = 1, 2$) liquid metal ion gun, Cs^+ sputtering gun and electron flood gun for charge compensation. Both the Bi and Cs ion columns are oriented at 45° with respect to the sample surface normal. The instrument vacuum system consists of a load lock for rapid sample loading and an analysis chamber, separated by the gate valve. The analysis chamber pressure is maintained below 5.0×10^{-9} mbar to avoid contamination of the surfaces to be analyzed.

(You can choose one or two of following options depending on your analysis)

For high mass resolution spectra acquired in this study, a pulsed Bi_3^+ primary ion beam at 25 keV impact energy with less than 1 ns pulse width was used. An electron gun was used to prevent charge buildup on the insulating sample surfaces. The total accumulated primary ion dose for data acquisition was less than 1×10^{13} ions/cm², an amount of ions which is within the static SIMS regime. The mass resolution on Si wafer is about $\sim 8000m/\Delta m$ at 29AMU.

For high lateral resolution mass spectral images acquired in this study, a Burst Alignment setting of 25 keV Bi_3^+ ion beam was used to raster a XXX μm by XXX μm area. The negative secondary ion mass spectra obtained were calibrated using C^- , O^- , OH^- , C_n^- , respectively. The positive secondary ion mass spectra were calibrated using H^+ , C^+ , C_2H_3^+ , C_3H_5^+ , C_4H_7^+ .

For depth profiles acquired in this study, 10 keV (or 3 keV) Cs⁺ with 20 nA current was used to create a 120 μm by 120 μm area, and the middle 50 μm by 50 μm area was analyzed using 0.3 pA Bi₃⁺ primary ion beam. The negative secondary ion mass spectra were calibrated using H⁻, C⁻, O⁻, C₃⁻, C₅⁻ and C₇⁻. (The calibration can be varied with different matrix).

Reference:

1. A. M. Belu, D. J. Graham, D. G. Castner, "Time-of-Flight Secondary Ion Mass Spectrometry: Techniques and Applications for the Characterization of Biomaterial Surfaces", *Biomaterials*, **2003**, 24, 3635 – 3653.