

SIMS Microanalyses for fO_2

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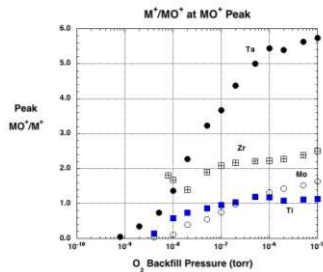
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Analyses of metal retrieved from experiments (either as capsule material or added fragments to the charge) may allow determination of oxygen fugacity during the experiment on a microscale, something John Holloway would have liked.

This technique builds on secondary ion mass spectrometry (SIMS) work [1], where U metal was sputtered with Ar^+ ions while backfilling the analysis chamber with oxygen. Energy spectra obtained on the sputtered species revealed large changes in the relative proportions of U^+ and UO^+ as a function of pO_2 and secondary ion energy. This suggested a potential relation between MO^+/M^+ and oxygen activity on the sample.



Examination of Ti, Zr, Mo, and Ta metals to date show varying responses of MO^+/M^+ to oxygen pressure (see Figure). For example, $MoO^+:Mo^+$ increases by a factor of >10 as the oxygen coverage increases by ~ 4 orders of magnitude. However, the TaO^+/Ta^+ ratio increases by $\sim 100x$ over the same range, while the TiO^+/Ti^+ ratio shows a $\sim 5x$ increase. The ZrO^+/Zr^+ ratio shows a complex pattern. Some of these ratios reach a nearly constant value at high oxygen coverage, which may indicate saturation of the surface.

Our results are not yet connected with absolute oxygen activity. If calibrated by metals equilibrated in gas mixing furnaces, this approach may allow use of oxide/element ion ratios to determine fO_2 in the charge, and possibly to determine how it changed during the run duration.

Applying this approach to samples synthesized at a range of conditions might be combined with other indicators of oxygen activity, such as Fe^{3+}/Fe^{2+} and other multivalent elements to create an optimized oxybarometer. Application to natural samples is in progress.

[1] Williams & Hervig (2000) In, *Secondary Ion Mass Spectrometry (SIMS)* **12**, 139-142.