New WiscSIMS IMS 1280 100 nm-Resolution Primary Beam Deflection System, for Accurate Aiming of Returned Samples.

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Introduction

- WiscSIMS IMS 1280: oxygen 3-isotope analyses with primary beam size as small as 1-2 µm:
  - Applied to the analyses of comet particles from Stardust missions and IDPs [1-4].
  - Heterogeneity of isotopic signatures of multiple µm-size mineral phases.
  - Accuracy of ion beam positioning is critical.
- Until now: positioning was limited by the optical resolution of the sample viewing microscope (originally ~3.5 µm but improved to 1.3µm using UV light optics [5]) and the step size of stage (1 µm).

➢ Nakashima et al. [3] developed a protocol to mark the grains of interest prior to SIMS analysis using a focused ion beam (FIB) to remove the carbon coat at the center of the target position. The absence of surface coating appears as a bright spot in the SIMS ion imaging.

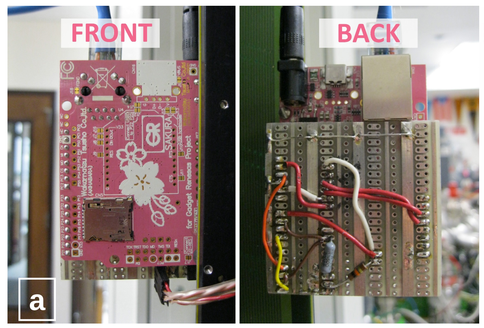
- Using this protocol, the primary beam is positioned on the sample surface with a significantly improved accuracy [3]. However, the aiming resolution (the difference between center of the primary beam and the center of a FIB mark) is curbed at ~0.4µm, because stage motion and primary beam deflection of the IMS1280 are limited to 1µm step.

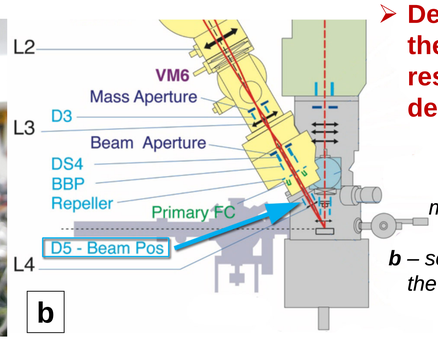
➢ **Therefore, we sought out to improve the aiming resolution of the IMS 1280**

NanoDeflector

- Modification of IMS 1280 scanning capability by adding a microcontroller board with X and Y D/A converters, adding signal to the last deflector of the primary column (D5).

➢ Custom C and Labview code on the microcontroller and the remote PC allow for user control.





➢ **Deflection and rastering of the primary beam at 0.1 µm resolution with maximum deflection of ± 50 µm.**

a *– front and back of Arduino compatible GR-SAKURA microcontroller with a precision D/A converter*
b *– schematic of the IMS 1280 showing the position of the D5 deflector on the primary beam column*

FIB-marking and aiming at small particles

- Marking of analysis point by a 1 µm² FIB-mark.

➢ **Dose of 0.4 nC/µm² (90s)**

- FIB mark is then imaged by SIMS and centered:

a – SEM SE image of FIB mark #10.

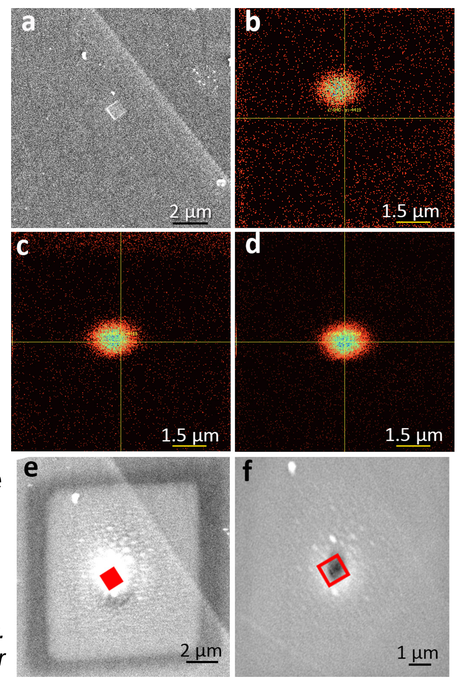
b – SIMS ^{16}O image before adjustment

c – after displacement of the stage

d – after adjustment with the NanoDeflector

e,f – SE image of the rastered area and the SIMS pit (~1.5 µm) after analysis, compared with the original position of the 1 µm FIB mark #10 (red square).

➢ **Aiming precision of 0.17 ± 0.15 µm**



a, e, f *– SEM Secondary Electron images.*
b, c, d *– SIMS ion images, 10 µm raster*

Comparison between stage and NanoDeflector displacements

- Nominal step distance: 4 um

a – by displacement of the stage.

➢ **3.6 ± 1.3 µm (2σ)**

Problem: too little displacement when direction changes (2.1 – 2.3 µm)

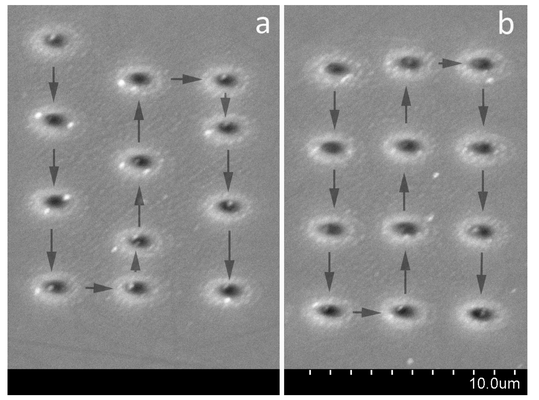
b – by deflecting the ion beam with the NanoDeflector

➢ **3.9 ± 0.38 µm (2σ)**

Problem: calibration of horizontal movement was too short. (3.5 – 3.6 µm)

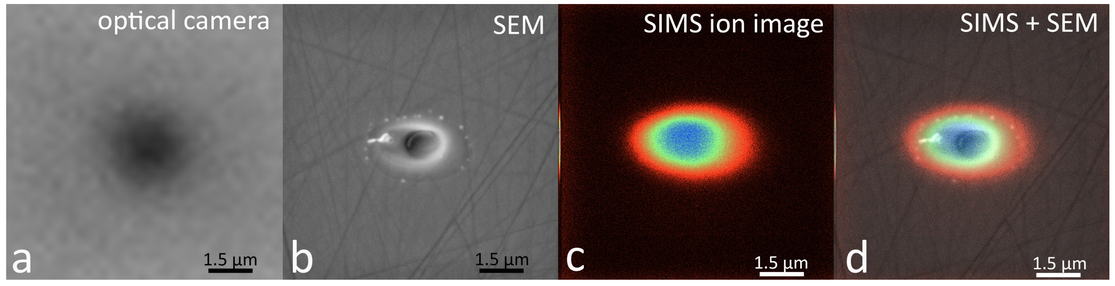
Average of displacement accuracy, excluding the specified issues:

➢ **3.9 ± 0.3 µm (2σ)** ➢ **4.0 ± 0.1 µm (2σ)**



SEM SE images of two series of pits. ***a*** *– by displacement of stage.* ***b*** *– by deflecting beam*

Comparison of imaging with SIMS and SEM



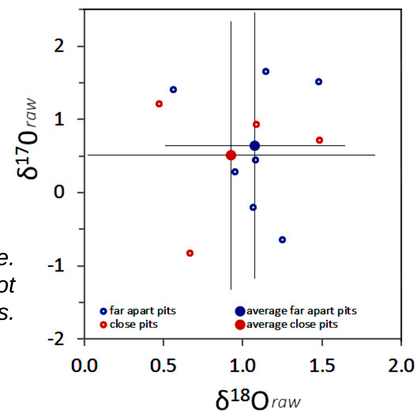
a *– Optical image with UV illumination (~1um resolution) of analysis pit* [5].
b *– SEM Secondary Electron image of the same pit.*
c *– SIMS ion images, 10 µm raster, of the same pit.*
d *– superimposition of images b and c shows that the pit corresponds to the blue and green area of the ion image. The red area is the Cs deposit.*

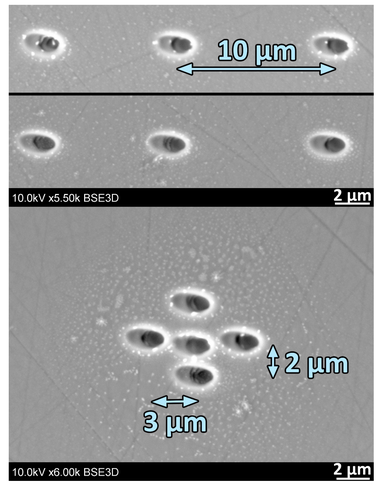
Precision for O 3-isotope analyses

- precision for ≈ 2 µm spots, in 2σ:
  $\delta^{18}O < 1$ ‰, $\delta^{17}O < 2$ ‰, $\Delta^{17}O < 2$ ‰

➢ No overlap when analysis points are as close as 3 µm apart horizontally and 2 µm vertically.

Analyses on San Carlos olivine. Results are given as raw data, not corrected for instrumental bias.





SEM SE images of widely separated pits (top) and close pits (bottom).

References: [1] **Nakamura T.** et al. (2008) *Science*, 321, 1664. [2] **Noguchi T.** et al. *EPSL*, 309, 198-206. [3] **Nakashima D.** et al. (2012a) *EPSL*, 357-358, p. 355-365. [4] **Nakashima D.** et al. (2012b) *Meteorit. Planet. Sci.* 47, 197-208. [5] **Kita N. T.** et al. *JAAS*, in press, doi: 10.1039/C4JA00349G