

5D-ToF-STIM Hyperspectral Imaging with a keV He⁺ Focused Ion Beam

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Scanning ion microscopy in combination with transmission ion energy-loss spectroscopy (IELS) for low-energy ions is a promising field of study, which seeks to provide complementary information to that of more conventional techniques like electron energy-loss spectroscopy (EELS) in scanning transmission electron microscopy (STEM). In contrast to electrons, ions are capable of both capturing and losing charge, allowing for charge exchange and neutralization processes [1]. In addition, while primary electrons are capable of exciting plasmonic and electronic states, as well as ejecting core-shell electrons, ions are further capable of temporarily merging their electron orbitals with those of the sample's atoms (potentially ejecting ion-induced Auger electrons), as well as ejecting sample atoms (measurable using mass spectrometry), creating unique signals not available to electron beams. The energy lost by the ions during these sample interactions can be measured as an increase in the time-of-flight (ToF) from the sample to the detector. Furthermore, the scattering pattern seen on the detector contains detailed information not only about the crystal structure, but also the trajectories of the ions within that structure [2]. By combining spatially resolved ion energy-loss and scattering information, we can thus create a rich 5D dataset: 2D position on the sample plane, 2D position on the detector plane, and finally ToF information for each ion or neutral collected. He⁺ ions are chosen for this work as they are particularly well suited to low-energy transmission ion microscopy due to their relatively small scattering cross-section (allowing for thicker samples) and their relatively low damage to the sample.

Our work at LIST focuses on low-energy (up to 30 keV) scanning transmission helium ion microscopy (STIM) coupled with ToF to analyze the angular and energy distribution of the transmitted helium ions and neutrals. Two prototype instruments are being developed: one based on a commercially available gas-field ion source (GFIS), which can achieve sub-nm probe sizes, and a second based on a commercially available DuoPlasmatron source with a probe size of <300 nm. Each prototype is equipped with a high-sensitivity microchannel plate (MCP) with a delay-line-detector (DLD), capable of measuring the exact location and timing of single ion or neutral impacts. However, due to the complexity of modifying the GFIS column to perform

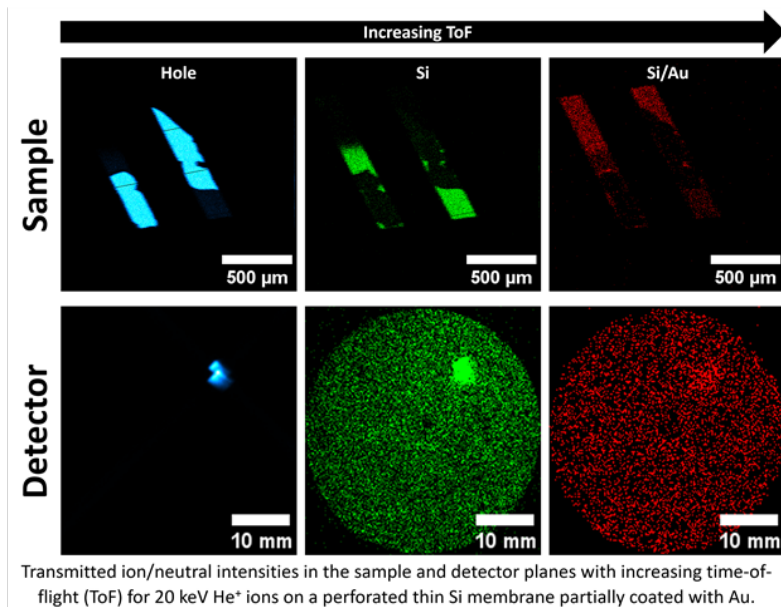
beam pulsing, it is not yet capable of TOF measurements (i.e. only capable of 4D datasets), while the DuoPlasmatron source comes with pulsing/blanking plates. The DuoPlasmatron based prototype also has a high flexibility in the instrument configuration, allowing for post sample ion deflection to determine the fraction of ions neutralized within the sample, as well as the ability to vary the post-sample flight distance.

Using a previous iteration of the DuoPlasmatron system, we have been able to create images by generating contrast from the total counts, the ratio of neutrals to ions, the scattering angle, and the energy loss (other criteria could also be envisioned with such a rich dataset). As each pixel in the sample image contains a full ToF spectrum and a scattering pattern, we have developed python software based on matplotlib and pandas with a graphical user interface for interactive plotting of sample images, detector images, and ToF spectra based on flexible combinations of ROI selections, in order to intuitively explore the 5D dataset. We have also compared our experimental scattering angle and energy-loss results from this instrument to SRIM simulations [3].

In parallel with the development of these instruments, we have also performed stationary-beam transmission ion experiments at the mature time-of-flight medium energy ion scattering (ToF-MEIS) beam line at Uppsala University, where extremely fast blanking speeds (below 1 ns) and higher accelerating voltages (up to 350 kV) are possible (Rev. Sci. Instrum. 83, 095107). Here we have seen differences in both the scattering pattern and ion energy distribution based on the direction of the beam passing through a thin single-crystal Si film with an amorphized layer on one side. In the case where the ions pass through the amorphous layer and then the crystalline layer, we observe a distinct crystalline scattering pattern, but a relatively broader energy distribution. For the inverted sample, where the ions instead pass through the crystalline layer first, we observe an apparently random scattering pattern, but a relatively narrower energy distribution. Ion channeling simulations performed with IMSIL [4] at TU Wien corroborate the dependence of both the energy and scattering distributions on the orientation of the sample with respect to the beam. These results are also in agreement with the simulation and experimental results of Holenak et al. demonstrating that a disordered surface layer can redirect incident ions into planes and channels, instead of randomizing their trajectories [5].

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Graphic:



Keywords:

Time-of-Flight, Ion Beams, Energy-Loss Spectroscopy

Reference:

- [1] R Holeňák et al., Vacuum 185, 109988 (2021).
- [2] H Krause et al., Phys. Rev. A 49, 283 (1994).
- [3] M Mousley et al., Microsc. Microanal. 29, 563 (2023).
- [4] G Hobler, Nucl. Instrum. Meth. B 96, 155 (1995).
- [5] R Holeňák et al., Phys. Rev. Applied 21, 024048 (2024).