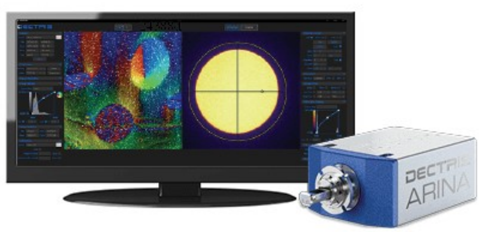
Cryo STEM of Low Melting Point Metals Enabled by Cryo FIB and EXLO

Michael Colletta, Jamie Ford, Joseph R Michael, Lucille A Giannuzzi, David A Muller

DECTRIS

ARINA with NOVENA

Fast 4D STEM



DECTRIS NOVENA and CoM analysis of a magnetic sample.

Sample courtesy: Dr. Christian Liebscher, Max-Planck-Institut für Eisenforschung GmbH.
Experiment courtesy: Dr. Mingjian Wu and Dr. Philipp Pelz, Friedrich-Alexander-Universität, Erlangen-Nürnberg.

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Microscopy and Microanalysis, **30** (Suppl 1), 2024, 1990–1991
https://doi.org/10.1093/mam/ozae044.985

Microscopy AND Microanalysis

Meeting-report

Cryo STEM of Low Melting Point Metals Enabled by Cryo FIB and EXLO

Michael Colletta[1], Jamie Ford[2], Joseph R. Michael[3], Lucille A. Giannuzzi[4,]*, and David A. Muller[1,5]

[1]*School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA*
[2]*Singh Center for Nanotechnology, University of Pennsylvania, Philadelphia, PA, USA*
[3]*Retired from Sandia National Laboratories, Albuquerque, NM, USA*
[4]*EXpressLO LLC, Lehigh Acres, FL, USA*
[5]*Kavli Institute for Nanoscale Science, Cornell University, Ithaca, NY, USA*

*Corresponding author: Lucille.Giannuzzi@expresslo.com

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Cryogenic techniques have significantly expanded the materials accessible for study in electron microscopy. The development of the cryo-focused ion beam (cryo-FIB) has enabled site-specific analysis in unique systems such as biological specimens, liquid-solid interfaces, and environmentally sensitive materials [1, 2]. Furthermore, this tool facilitates the implementation of specimen preparation techniques for high-resolution characterization with cryogenic scanning/transmission electron microscopy (cryo-S/TEM). Cryo-FIB lift-out specimen preparation is a powerful technique for researching these systems. However, successful *in situ* specimen manipulation is often challenging due to issues with specimen attachment, thermal stability, and the structural integrity of the specimen [3]. The cryogenic *ex situ* lift out (cryo EXLO) technique has recently been developed to address these challenges and has successfully been applied to both soft matter organic polymers and biological vitreous materials [4]. In this work, we demonstrate, through a combination of cryo-FIB thinning and EXLO manipulation, the preparation of high-quality, electron-transparent specimens of low-melting-point indium metal (mp=156.6°C).

Recent studies have revealed that the issues associated with room temperature milling conditions in indium metal, leading to void formation, cannot be solely attributed to reactivity with the Ga^+ ion beam, as voids form irrespective of the FIB ion used (e.g., Ga+ or Xe+) [5]. Rather than the ion source, this milling issue is associated with fundamental ion-solid interactions, where low melting point materials typically exhibit higher FIB milling sputter yields. Previous work has also shown that the sputter yield of a material increases as a function of temperature once it reaches ~70% of its absolute melting point [6]. We also have evidence of increased FIB milling sputter yields as a function of temperature. With Xe FIB milling, the sputter yield increases to ~ 20% when milled at a stage temperature of 25°C compared to 55°C.

For this reason, to properly address proper milling conditions in indium metal, cryogenic temperatures are necessary. With the indium metal cooled to -175°C, we can achieve thin, electron-transparent specimens without noticeable void formation (Figure 1a). The stage is then warmed back to room temperature and specimen manipulation to a TEM grid is accomplished with EXLO manipulation. This grid is transferred to a S/TEM, where it is cooled back to liquid nitrogen temperatures for cryo-STEM imaging (Figure 1b). In STEM, we achieved atomic resolution of the indium crystal structure, indicating the specimen was sufficiently thinned and free from significant surface contamination with final 5 kV milling (Figure 1c). This work presents an effective method for the preparation and high-resolution study of challenging, low-melting-point metals through a combination of cryo-FIB and EXLO techniques [7].

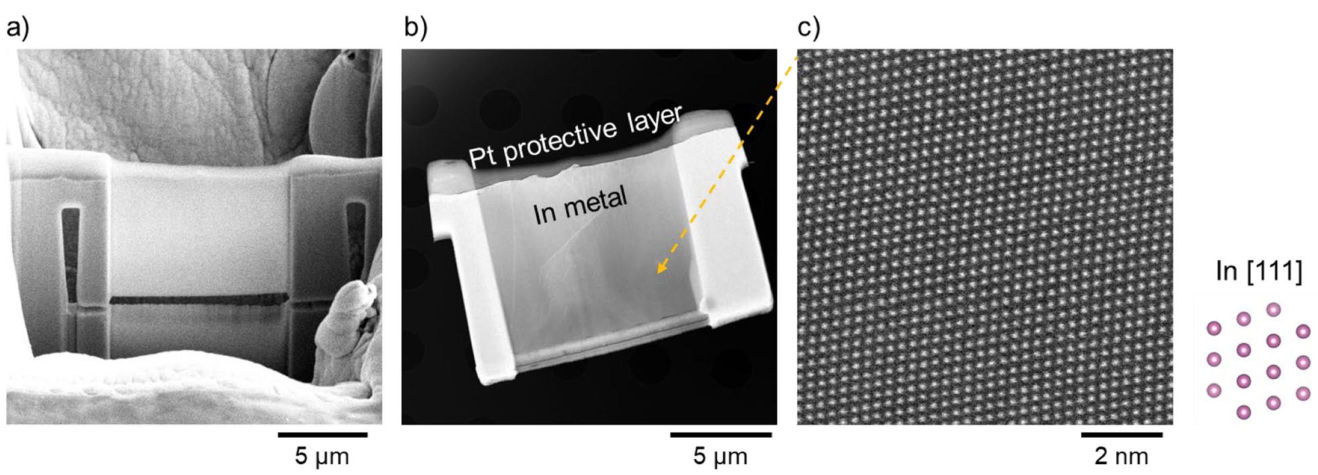

Fig. 1. a) SEM image of cryo-FIB thinned indium metal specimen. b) Cryo-STEM HAADF overview showing successful transfer to TEM. c) Atomic resolution of indium [111] lattice demonstrates quality of FIB specimen.

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7. The authors acknowledge funding from DOE BER award DE-SC0020511. This work also made use of the Cornell Center for Materials Research Shared Facilities which are supported through the NSF MRSEC program (DMR-1719875).

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