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Development of a Stable Low Temperature Sample Holder for the Side-Entry Transmission Electron Microscope

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The spatial resolution and flexibility of the Scanning/Transmission Electron Microscope (S/TEM) as a characterization tool is unmatched. Atomic resolution imaging is quickly becoming an essential component in the characterization workflow. The experimental capabilities of the side-entry S/TEM are also readily augmented by the addition of specialized sample holders, which can subject the sample to various conditions *in situ*. Holders for the S/TEM can expose samples to heating, cryogenic conditions, gases, liquids, and mechanical strain [1]. Most of these holders are available in stable configurations suitable for high resolution imaging.

A notable deficiency in the available catalog of designs are stable holders optimized for temperatures below room temperature but above the boiling point of common cryogens. Cryogenic sample rods can be fitted with a heating unit to access intermediate temperatures but doing so often causes excessive boiling of the cryogen. This boiling introduces instability which limits the achievable spatial resolution while using the holder. The only notable exception to this is Ref. [2], which has recently reported sub-angstrom resolution in this temperature range by isolating the boiling cryogen from the sample rod. Rather than dampening vibrations with mechanical decoupling, this study details a novel approach to access the low temperature region using thermoelectric cooling, effectively eliminating vibrations altogether.

A low temperature sample rod prototype has been designed using thermoelectric cooling. The thermoelectric element may be used to raise or lower the temperature of the sample by reversing the direction of heat flow. Heat from the sample is rejected into a heat sink containing a frozen coolant, keeping the "hot side" of the thermoelectric system fixed at the coolant's melting point. Initial testing has proved that the prototype can sustain a temperature of 233 – 323K at the sample.

References:

[1] "TEM Specimen Holders", https://www.gatan.com/products/tem-specimen-holders (accessed Feb. 12, 2022).
[2] B. H. Goodge, E. Bianco, N. Schnitzer, H. W. Zandbergen, and L. F. Kourkoutis, "Atomic-Resolution Cryo-STEM Across Continuously Variable Temperatures," *Microsc. Microanal.*, vol. 26, no. 3, pp. 439–446, Jun. 2020, doi: 10.1017/S1431927620001427.
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