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In-situ cryo-biasing heating TEM sample holder with full-range temperature control from -170°C up to >1000°C

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Background

Traditionally in-situ TEM biasing, heating and cooling have been split capabilities, requiring different TEM holders. In-situ cryogenic cooling and biasing of specimens during scanning/transmission electron microscopy (S/TEM) has previously enabled the in-situ characterization of various quantum interfaces and chemical phase interactions in battery materials and other strongly correlated systems [1]. Quantum material responses must be studied under cryogenic conditions because many of the relevant properties in these materials only manifest below certain temperatures, while battery material interfaces are sensitive to electron beam damage when not cooled.

Combined in-situ sample heating with biasing has only become available in recent years with the use of the chip-based experimental platform, which has enabled capture of high-temperature microstructural and microchemical evolution alongside measurement of high-temperature electrical response in materials systems. Now, with the inclusion of on-chip biasing and thin film heater chip designs, we have developed a novel in-situ cryo-biasing heating TEM holder which enables simultaneous electrical stimulus and imaging of a sample across the full temperature range from cryogenic temperatures to over 1000 °C, while matching room temperature (RT) microscope spec for resolution and drift. The basic functionalities of heating, cooling, biasing, and imaging are demonstrated alongside discussion of different applications.

Methods

The TEM sample holder has an attached LN2 dewar that cools the sample down from RT to the lowest operating temperature (< -170°C) and can be controlled via software with closed-loop feedback at any temperature up to RT. Below RT where on-chip temperature measurements become increasingly inaccurate, precise temperature control is maintained using a conventional resistance heater and miniature thermocouple at the sample in the TEM holder tip. Nine electrical contacts to the user's sample or device allow on-chip biasing. Biasing experiments can now also be extended above RT to >1000°C using thin film sample heating with reliable on-chip temperature sensing. Since the heated area is small, the response time to temperature changes is fast and sample drift is minimized across the entire temperature range, resulting in image stability that matches the RT performance. Here, we present an example of a battery process after cooling a single nanowire system from RT down to liquid nitrogen temperature ~ -170°C [2].

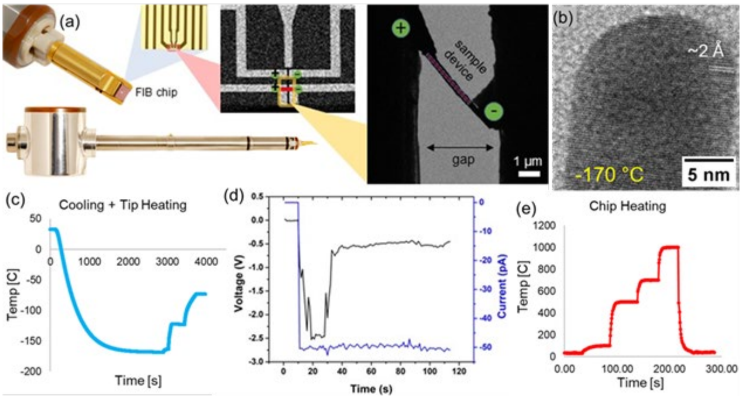
Results

In Fig 1a, electrical biasing was performed on a nanowire sample bridging the electrodes on a biasing chip using this holder at RT down to near-liquid nitrogen temperature to protect damage in these beam-sensitive materials [2]. With constant current applied at cold temperatures, the voltage drops across the nanowire as a reaction proceeds with the plating of a dendrite layer on the surface, as shown in Fig 1d. In Fig 1b, atomic lattices are shown as a demonstration of resolution. Temperature control of the cooled and heated ranges is also shown in Fig 1c and 1e, respectively. The figures exhibit the core functionalities of the tool: stable and reliable sample biasing, heating, and cooling under atomic resolution TEM.

Conclusions

The In-situ cryo-biasing heating TEM sample holder presented here enables, for the first time, simultaneous electrical stimulus and high-resolution imaging of a sample across the full temperature range, from cryogenic up to high temperatures. This instrument will accelerate the development of the next generation of electronic, quantum, and energy storage materials devices. Combination of the two temperature regimes expands the range of temperatures available for dynamic temperature experiments, wherein multiple processing, imaging, or synthesis steps must be performed at various temperatures. High-resolution in-situ observation of operating (solid-state) battery interfaces in their full operational range of -40°C to 80°C is traditionally difficult to achieve due to air sensitivity, electron beam damage at RT, and lack of temperature control in TEM biasing platforms [3-5]. With this new tool, batteries may now be electrochemically cycled in situ at their entire operating range and then lowered to cryogenic temperature for imaging to combat electron beam effects, without the need for sample transfer between tools. With increasing demand for batteries that function at high temperatures, quantum material responses, synthesis, and processing, and material phase information from cryogenic to high temperature, the cryo-biasing TEM holder will provide the expanded versatility required of temperature-controlled in-situ electrical biasing systems.

Graphic:



Keywords:

cryogenic, heating, transmission electron microscopy

Reference:

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