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In-situ Thermal Testing on Nanostructures in TEM

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As a long-standing problem, sample temperature under electron irradiation in TEM [1] is critical for both scientific interests and technological applications. While, due to the extremely small area of interest, robust method for probing temperature at micron/nano scale is still missing. Efforts were made to measure the temperature rise of the TEM sample, by using carbon nanotube filled with Ga [2], small particles with low melting point [3], MEMS device [4] and EELS spectrum [5]. Here, we demonstrate a new type of microthermometer based on a VO_2 nanowire (NW), which derives its fundamental sensing mechanism from the well-known metal–insulator transition in VO_2 at 68 °C [6]. Using this new type of microthermometer, we can not only measure the temperature of small structures but also the heat flow rate generated by a local interaction with the NW, such as the bombardment of high-energy particles.

As shown in Figure 1a, a 59.8-mm-long VO_2 NW with a cross-sectional area of 0.20 mm^2 was transferred onto a Cu TEM grid using a micromanipulator in a focused ion beam (FIB). Following this, a 34.5- mm-long Si NW with a cross-sectional area of 0.31 mm^2 was mounted on top of the VO_2 NW. At the joints of both the TEM grid and VO_2 NW and the Si NW and VO_2 NW, electron beam induced Pt was deposited to join them together, as shown in Figure 1b schematically. Owing to the vacuum of the TEM column, heat convection can be ignored and radiation contributions are also safely negligible. Therefore, our analysis can be limited to one-dimensional thermal conduction. The temperature measurement concept is then based on the fact that as more and more electrons are focused on the extended part of the VO_2 NW, the temperature of the local region becomes higher and higher, until the metal–insulator transition (MIT) occurs at 68 °C. The phase transition can be clearly observed in diffraction patterns of the pristine monoclinic I phase (M1) VO_2 and the phase transformed rutile phase (R) VO_2 at high temperature. The heat flows from the higher temperature part of the NW (under the electron beam) to the lower-temperature part (attached to the TEM grid), resulting in a temperature distribution shown schematically in Figure 1c. When the electron beam is moved onto the Si NW as shown schematically in Figure 1d, a phase transition in VO_2 was still expected, but the MIT in VO_2 NW could not be observed directly. On the basis of the previous research [7], it is known that the MIT induces a structural change corresponding to a shrinking of the NW by about 1%. Here, this structual change could be seen as an abrupt lateral jump of the Si and was used as an indication of the MIT. Figure 1d shows the schematic temperature distribution when a high-enough electron beam current was focused onto the Si NW to heat the Si NW–VO_2 NW joint to 68 °C. As shown in Figure 2, about 1 to 3 μW, and 0.62 μW of heat is deposited by the incident electron beam into the VO_2 NW and Si NW, respectively. Owing to its small size the vanadium dioxide nanowire-based microthermometer has a large measurement range and high sensitivity, making it a good candidate to explore the temperature environment of small spaces or to monitor the temperature of tiny, nanoscale objects.

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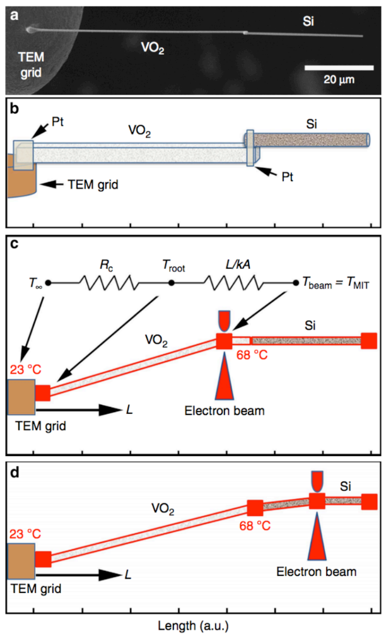

Figure 1. Temperature measurement using a VO_2 and Si nanowire in a TEM. (a) Scanning electron microscope picture of the VO_2 NW cantilever, extending from the TEM grid, on top of which a Si NW was mounted. (b) Schematic of the setup in a, showing the location of the electron beam-deposited Pt joints. (c) Diagram of the thermal resistance from the TEM grid to the region under the electron beam and schematic of the temperature distribution within the NWs, when the electron beam was located on VO_2 NW. (d) Schematic of the temperature distribution within the NWs when the electron beam was located on Si NW.

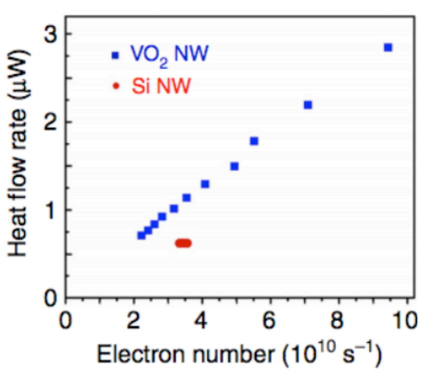

Figure 2. Experimental measurement of heat flow through Si and VO_2 nanowires