

Quantification of Electron Beam Heating Effect in TEM

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Electron beam heating effect is inevitable for specimens under irradiation in TEM, which gives rise to a temperature increase [1]. When encountering a target, fast electrons interact strongly with both atomic nuclei and atomic electrons in a Coulombic manner. Since the energy directly transferred to the atomic nuclei is obviously smaller than the one to atomic electrons, most of the energy loss in interacting with the specimen is attributed to the atomic electron excitations. Consequently, the average rate of the energy loss can be calculated from the Bethe-Bloch expression [2],

$$-\frac{dU}{dx} = \frac{8\pi a_0^2 U_R^2}{mc^2 \beta^2} \left\{ \ln \frac{mc^2 \beta^2 U}{2(T_{\min})^2 (1-\beta^2)} - (2\sqrt{1-\beta^2} - 1 + \beta^2) \ln 2 + (1-\beta^2) + \frac{1}{8}(1-\sqrt{1-\beta^2})^2 \right\}$$

Here, a_0 is the Bohr radius, U_R is the Rydberg energy, m is the electron mass, $\beta=v/c$, T_{\min} is the minimum transferable energy. The major portion of the energy loss eventually ends up as heat. Given a known specimen geometry, the local temperature rise can be estimated theoretically. The TEM specimen temperature is often difficult to measure locally in practice, so that the experimental investigation is very limited to understand the electron beam heating effect.

Here, we show results of quantifying electron beam heating effect in Si nanowires and VO₂ nanowires, by using the metal-insulator transition (MIT) in the VO₂ nanowire as an effective nanothermometer [3]. The experimental setup is shown in the Figure 1 (a), in which case the Si nanowire was transferred onto the top of the VO₂ nanowire. FIB deposition of Pt was employed to bond the Si-VO₂ joint and the VO₂-Cu grid joint. To evaluate the influence of the beam-specimen interaction distance, the VO₂ nanowire was tilted along the longitudinal axis as shown schematically in Figure 1 (b). Therefore, the effective interaction thickness varied from about 170 nm to 250 nm. Figure 2 (a) shows the electron beam flux as a function of the traversed VO₂ thickness; local heating of the specimen to 68 °C [4] is detected via the VO₂ MIT at that temperature. The phase transition was confirmed in corresponding diffraction patterns in Figure 1 (c) and (d). This shows clearly that increasing the beam-sample interaction length makes it easier to raise temperature with lower electron flux, which is consistent with the Bethe-Bloch expression. No other obvious specimen orientation effect was noticed. More importantly, the empirical equation, $Q=Q_0 \cdot (t/\lambda) \cdot N_e$, was proved to successfully describe the electron beam heating effect in TEM, where the Q_0 is the energy stored in the specimen in each collision event, t is the specimen thickness, λ is the mean free path of scattering, N_e is the electron beam flux. A material-dependent parameter, Q_0/λ , was inferred to be $2.67 \times 10^{-14} \pm 1\%$ mW/nm for VO₂ and $2.08 \times 10^{-14} \pm 1\%$ mW/nm for Si, respectively. Heat generated by the electron beam heating effect in such materials could be estimated with such parameter in any TEM working at 300 keV, which is based on the experimental measurement. These results open an avenue to quantitatively investigate electron beam heating effects in solid materials experimentally, complementary to the theoretical estimates based on the Bethe-Bloch expression.

References:

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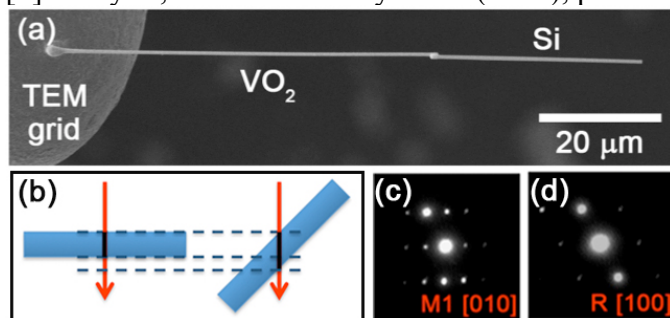


Figure 1. (a) SEM image of the experimental setup for heat measurement in VO₂ and Si nanowire in TEM. (b) Schematic of effective interaction thickness of a tilted VO₂ nanowire. (c) Diffraction pattern of the M1 phase VO₂ at ambient temperature. (d) Diffraction pattern of the R phase VO₂ at elevated temperature.

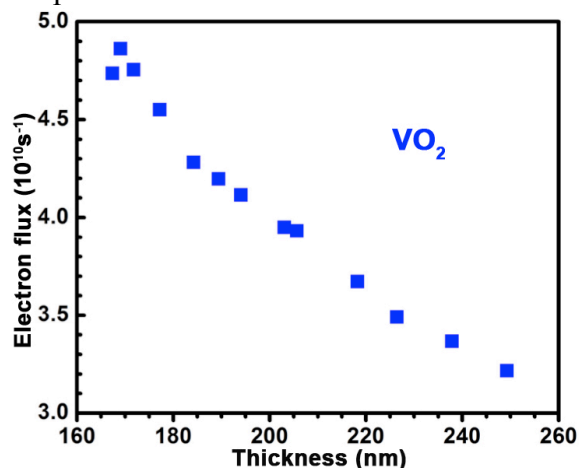


Figure 2. Electron flux as a function of the effective thickness of VO₂ nanowire corresponding to triggering the MIT phase transition at 68 °C under the electron beam.