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Wetting -Dewetting Transitions of Ultrathin Nickel Films Deposited on Silicon (100) Substrates

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The transition of continuous films that wet a substrate surface to a non-wetting or dewetted state have been extensively studied in the literature for film thickness in the micrometer range [1-3]. Dewetting is a diffusion driven process, during which the system decreases its free energy by minimizing the interfacial area between film and the substrate. Continuous shrinking of device structures demands the investigation of solid-state wetting -dewetting transitions for ultrathin films at temperatures well below the melting point.

Here, we report results of cross-sectional aberration corrected STEM, HRTEM, and EELS studies of solid-state dewetting. In-situ heating during HRTEM and STEM observations was used to induce wetting-dewetting transitions of Ni films on silicon.

Thin nickel films with nominal thicknesses of 6nm and 17nm were deposited on the (100) surface of silicon by DC magnetron sputtering. Deposition rates of 2.7 nm/sec and 11nm/sec were used, respectively. Dilute hydrofluoric acid was used to remove any native oxide layer on the silicon substrate prior to deposition. Sputtering occurred at room temperature, at a base pressure of 1.8×10^{-8} torr. Cross-sectional TEM specimens of the as-deposited films were prepared by standard cutting, grinding, dimpling and ion-milling techniques.

HRTEM observations of the as-deposited films (prior to in situ annealing) revealed the formation of multiple distinct layers parallel to the substrate surface (Figure 1). EELS spectrum images recorded from the as-deposited 17nm thick film show the formation of nickel silicide reaction layers with a gradually changing stoichiometry ranging from nickel-rich silicide over NiSi to silicon-rich silicide close to the film/substrate interface [4]. The thinner film reveals chemically discrete layers. For both film thicknesses a SiO_x ($x \leq 2$) layer is observed that formed due to the Kirkendall effect.

In-situ heating experiments have demonstrated that it is feasible to observe wetting-dewetting transitions by cross-sectional TEM (see Figure 2). The thicker films showed a transition temperature of 575°C, while the 6nm thin film de-wet 400°C. $NiSi_2$ was observed to form from both layered structures during the annealing process for temperatures well below the bulk transition temperature of 850°C.

The silicon oxide layer formed during film deposition plays a critical role for the formation of the layered structures and ultimately serves as a diffusion barrier for Ni during the wetting-dewetting transition. Detailed investigations about the formation of the oxide layer and the atomic and electronic structure of the corresponding interfaces are currently underway.

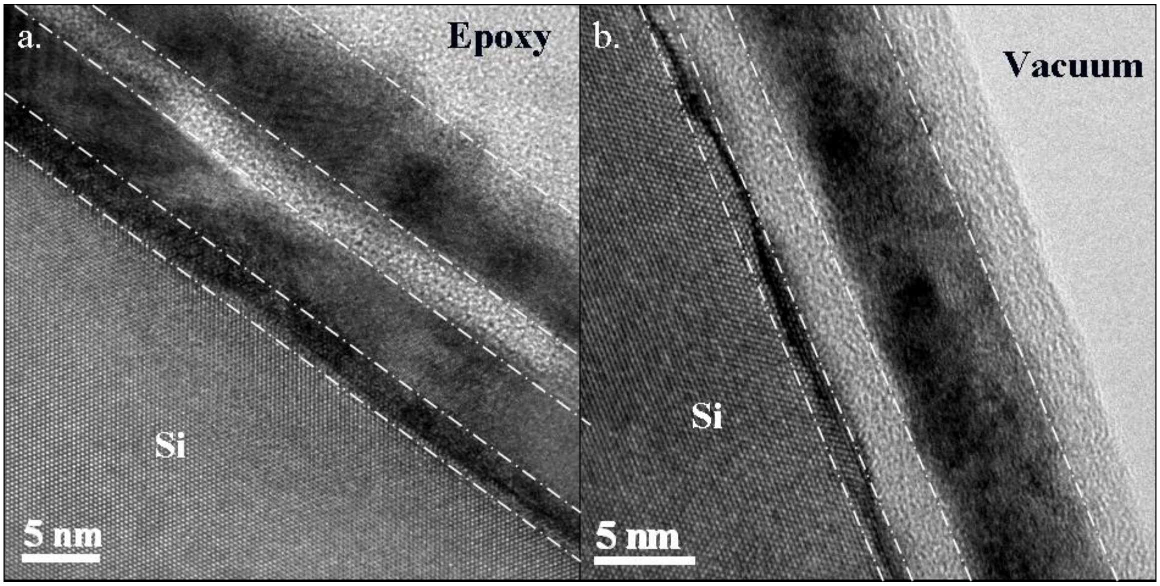

Figure 1. HRTEM micrographs of the as-deposited films. (a) 17nm Ni film and (b) 6nm Ni film.

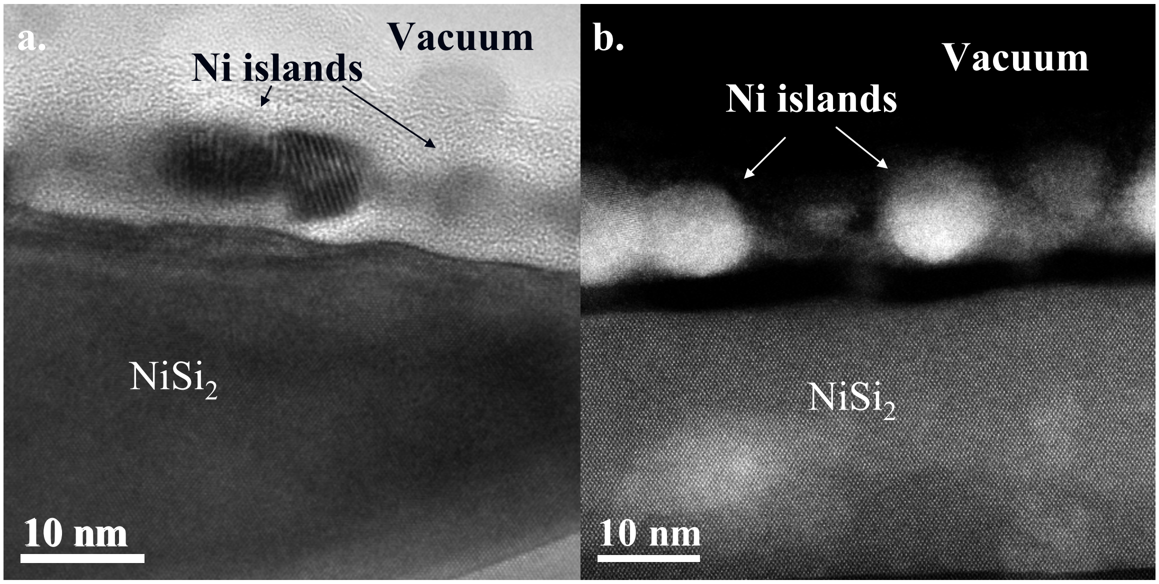

Figure 2. (a) HRTEM micrograph of the 17nm Ni film after annealing at 575°C. (b) HAADF image of the 6nm film after annealing at 490°C.

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