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# In-situ Heating Investigations of Dewetting Transitions in Ultra-Thin Ni Films on $SiO_2$ Layers

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Solid State dewetting occurs when an imbalance between interface and surface free energies occurs for thin films on solid substrates. Solid state dewetting is initiated by grain boundary grooving, where grain boundaries in the thin film meet the free surface [1]. Ridge formation occurs on the surface of the film by the diffusion of atoms away from the grain boundary-free surface triple point, further driving the nucleation and growth of holes. Previous in-situ studies successfully measured the rate of nucleation and growth of holes during the dewetting process, but none have observed the progression of thin film morphologies in real-time [2, 3]. A previous study has shown the Ni-Si reaction will compete with the Ni dewetting transition for Ni films deposited on native $SiO_2$ layer[4]. The aim of this study is to isolate, and directly observe the dewetting of ultra-thin Ni films on thermally grown $SiO_2$ layers. We use existing in-situ heating techniques for observation of the dewetting transitions inside the Transmission Electron Microscope (TEM). Electron Energy Loss Spectroscopy (EELS) and aberration corrected Scanning Transmission Electron Microscopy (STEM) is used to characterize the thin films before and after dewetting.

Thin nickel films with nominal thicknesses ranging between 3nm and 30nm were deposited on $SiO_2$ films by DC magnetron sputtering. The $SiO_2$ films were thermally grown on silicon (100) substrates. Before the deposition of Ni, organic material is cleaned from the surface of the $SiO_2$ films with a UV/Ozone machine. Room temperature, DC magnetron sputtering of nickel films was carried out at rates of 0.19Å/sec and at a base pressure of $1.8x10^{-8}$ Torr. Cross-sectional TEM specimens of the as-deposited films were prepared by wedge polishing and ion milling [5], as well as by Focus Ion Beam (FIB) sectioning.

In this study we isolated the Ni dewetting transition by depositing Ni films on 12nm thick $SiO_2$ layers. Fig. 1a shows a high-angle annular dark field STEM micrograph of the as-deposited interface configuration. EELS lines scans were acquired across the $Ni/SiO_2$ interface, confirming that Ni does not diffuse through the SiO2 layer and react with the Si substrate (Fig. 1b). In-situ annealing experiments revealed that the dewetting behavior varies throughout the Ni film. In some areas the film remained continuous (Fig. 2a), while in other areas island formation occurred (Fig. 2b). Cr and the formation of graphitized carbon were observed on the Ni islands as well as at the $Ni/SiO_2$ interface. Currently we are investigating whether Cr and graphite formation contributed to the varying behavior of the Ni film as observed in Fig. 2.

To further develop the in-situ annealing technique, a method is being developed to place a thin, cross-sectioned lamella onto a Protochips Aduro device using a dual beam FIB instrument. Preliminary results show that placement of a thin lamella onto the Aduro device is possible. First results from the new Protochips double-tilt Aduro heating holder will be reported during the presentation.

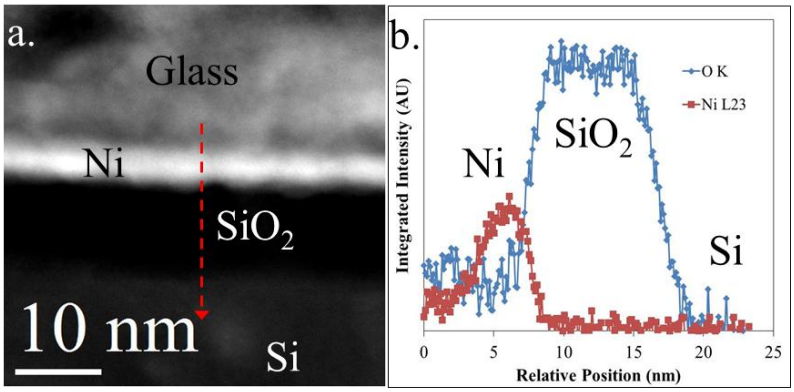


**Figure 1.** (a) HAADF micrograph of the as-deposited 3nm thick Ni film(b) EELS integrated intensities from the O K-edge and the Ni $L_{2,3}$ edge plotted as a function of distance across the red dotted line in (a).

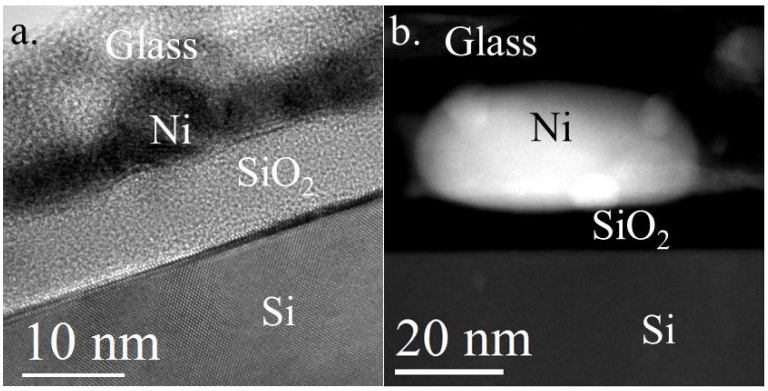


**Figure 2.** The 3nm Ni film after in-situ annealing (a) HRTEM micrograph of a continuous area of the film (b) HAADF micrograph of an area where a Ni island has formed.

[1] E. Jiran, C.V. Thompson, CAPILLARY INSTABILITIES IN THIN-FILMS, Journal of Electronic Materials, 19 (1990) 1153-1160.
[2] R. Felici, N.M. Jeutter, V. Mussi, F.B. de Mongeot, C. Boragno, U. Valbusa, A. Toma, Y.W. Zhang, C. Rau, I.K. Robinson, In situ study of the dewetting behavior of Ni-films on oxidized Si(001) by GISAXS, Surface Science, 601 (2007) 4526-4530.
[3] E. Jiran, C.V. Thompson, CAPILLARY INSTABILITIES IN THIN, CONTINUOUS FILMS, Thin Solid Films, 208 (1992) 23-28.
[4] A.M. Thron, P.K. Greene, K. Liu, K. van Benthem, Structural changes during the reaction of Ni thin films with (100) silicon substrates, Acta Materialia, 60 (2012) 2668-2678.
[5] P.M. Voyles, J.L. Grazul, D.A. Muller, Imaging individual atoms inside crystals with ADF-STEM, Ultramicroscopy, 96 (2003) 251-273.
[7] The authors acknowledge financial support through the National Science Foundation (DMR-0955638).

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