FIB Cross-Sections for Morphological Analysis of Ni-P Hard-Mask Transformation during Plasma Etching

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Ni layers are frequently used as hard masks for deep plasma etching of Si in MEMS-NEMS fabrication with etch selectivity as high as 10^2-10^3.[1-4] For Ni layers deposited by e-beam film evaporation, mask hardening (gradual reduction of Ni removal rate) during high-density (ICP) plasma etching was observed, resulting in growing Si/Ni etching selectivity.[1] As an alternative, electroless deposited Ni-P films[2] were also employed as hard masks for high-density deep reactive ion etching using alternating $(Ar+SF_6)/(Ar+C_4F_8)$ gases for etching /passivation steps (Bosch-like process). A similar effect of gradual decrease of Ni-P mask removal rate during the etch process was observed here, and a morphological cross section analysis with focused ion beam revealed a formation of a fluorocarbon (FC) layer over the Ni-P film, confirmed also by XPS analysis. Moreover, after an initially uniform deposition of a few nm thick FC layer (likely to be the reason of decreasing Ni mask removal rate), formation of islands and then well pronounced FC-hillocks on the surface was revealed using FIB-cross-sectioning. This happens probably due to increased mobility and charging of a FC layer under energetic $CxFy^+$ ion bombardment.

Ni-P film (~1.9 µm thick), was grown over high-purity <100> n type Si wafers in two steps. First, the wafer was dipped in a Pd surface activation solution and after, the Ni-P film was grown in a chemical bath. The film composition is Ni: 80%, P: 14.9%, Pd: 0.2%, Pb: 0.6%, obtained with RBS analysis. The wafer with the Ni-P film was processed with 120, 240 and 360 steps of 10s/10s of etching/passivation with $Ar+SF_6$ and $Ar+C_4F_8$ gases, respectively.

SEM images of a mask surface before and after processing (Fig. 1), show development of surface roughness in a specific form, eventually resulting in a formation of hillocks. FIB cross-sectioning was done to reveal that the roughness development happens basically not due to non-uniform Ni removal as could be expected, but because of gradual growth of a FC layer, that after certain stage starts a global restructuring over the sample surface forming well pronounced hillocks with very sharp nanometer-scale tips (Fig. 2). The underlying mechanisms and possible applications of the observed effects are discussed.

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1 A. M. Nunes et al, ECS Transactions, 14 (1) (2008), p. 403.
2 V. M. Dubin et al, J. Electrochem. Soc., 139 (5) (1992), p. 1289.
3 M. Parameswaran et al., J. Electrochem. Soc, 140 (7) (1993), p. L111.
4 B. Elsener et all, J.Appl. Electrochem., 38 (2008), p. 1053.

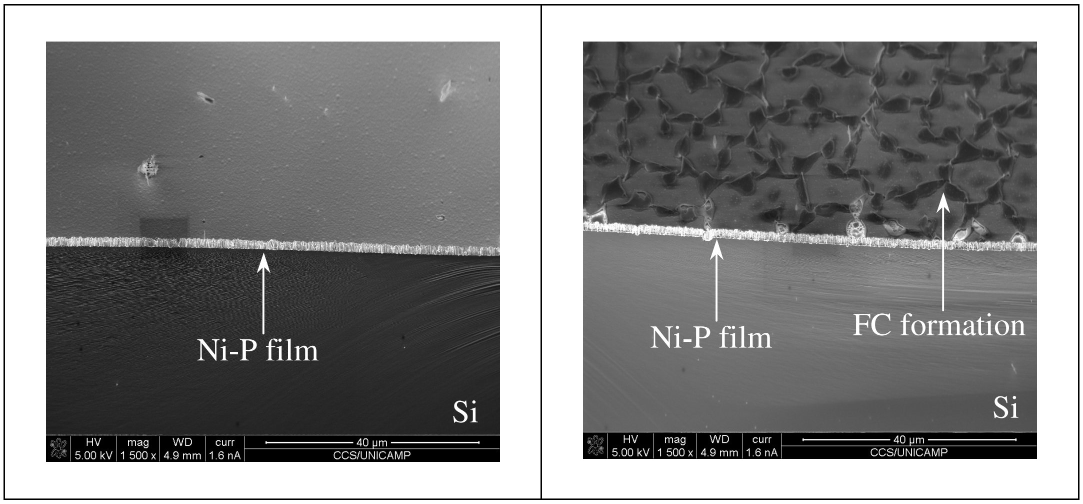

Figure 1. SEM images of Ni-P surfaces, before (left) and after (right) processing.

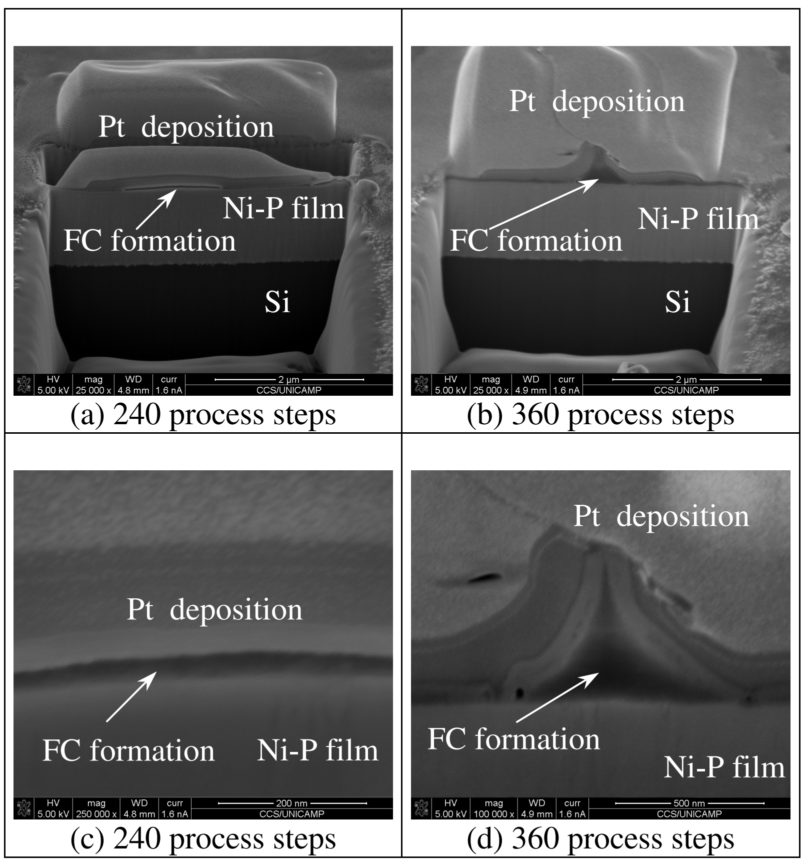

Figure 2. FIB cross-section analysis. (a) and (b): Pt protective layer local deposition (FEB followed by FIB) and FIB milling. (c) and (d): higher magnification images for FC film morphology characterization.