A comprehensive EELS/EFTEM study of focused ion beam prepared samples of nitride- and oxide thin films

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Thin nitride- and oxide films have found many important applications due to their favorable thermal and electric properties, their hardness and wear resistance, and for instance have led to new computer chip designs as improved nitride diffusion barriers became available. In these studies, nitride- and oxide films grown by rapid thermal processing (RTP) and by pulsed laser deposition (PLD) have been subjected to a comprehensive transmission electron microscopical study. In previous studies [1-3] it was shown that TEM and, in particular, EELS and EFTEM are especially suitable tools for the characterization of multilayer structures on the nanoscale. The samples under investigation on the one hand have been vanadium nitride/oxide thin films on a silicon dioxide/silicon substrate prepared by RTP and on the other hand Mg/Ti/Zr/Zn-oxide multilayers on a silicon substrate processed by PLD. The layers in the selected RTP-sample were obtained by sputter-coating the substrate with vanadium, then annealing the sample in a NH₃-atmosphere at 700°C for nitridation, whereas the PLD sample was a multilayer film made up laser-ablated Mg-Ti-Mg-Zr-Zn-oxides (sequence as stated) at rather low temperatures (20-50°C) with individual layer thicknesses below 150nm. By using a dual beam focused ion beam instrument (FEI Nova™ 200) uniformly thin specimen thicknesses of around 50nm, suitable for EELS and EFTEM investigations, could be prepared. In a final preparation step, the cross-sections have been treated with a 5kV ion beam to remove possibly damaged surface material and to obtain parallel TEM lamellae. For TEM investigations, a Philips CM20 (200kV, LaB₆, GIF, ~1eV energy resolution) and FEI Tecnai™ F20 with a WIEN-filter monochromator (200kV, Schottky-FEG, HRGIF, ~0.2eV energy resolution) were used. Electron energy loss data were acquired in the form of monochromated and conventional EELS spectra as well as spectrum images and as extracted elemental maps. By involving post-processing steps like interactive correlation partitioning, factorial filtering via principal component analysis and comparison of the monochrome EELS data with calculations, interesting information about the layer chemistry could be obtained.

References