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On search for hidden experimental variables during thin film growth by high-vacuum magnetron sputtering

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Surface properties of refractory ceramic transition metal (TM) nitride thin films grown by magnetron sputtering are essential for resistance towards oxidation necessary in all modern applications. Unfortunately, the effects of or conditions for residual gas exposure, venting strategy, and the long-term storage are not explicitly addressed in the literature. We seek to change the status quo by investigating the role of venting temperature T_v, a “hidden” experimental variable often not considered and reported, but, as we show here, defining the surface chemistry of the TiN layers. We employ the previously developed Al-cap technique[i] to separate the effects of residual gas exposure in the high-vacuum environment during the post-deposition phase from those introduced during the following venting sequence and air exposure. With the help of x-ray photoelectron spectroscopy (XPS) analyses performed on a series of TiN samples as a function of T_v we find that majority of surface reaction products, including TiO_2, TiO_xN_y, and N_2 previously detected after prolonged annealing experiments, form shortly after vent, provided that T_v is sufficiently high. This has implications for all sorts of practical studies where the surface composition of TM layers is assumed to be fixed once the same growth protocol is used. We show that this is definitely not the case for the TiN model materials system, and that the venting temperature has a substantial effect on the composition and thickness-evolution of the reacted surface layer and should therefore be reported. [i] G. Greczynski, I. Petrov, J.E. Greene, and L. Hultman, J. Vac. Sci. Technol. A 33 (2015) 05E101

Keywords
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