

Characterization of Molybdenum Oxide: Understanding Growth Kinetics and Molybdenum Consumption for Materials Science Applications

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The natural tendency of molybdenum to oxidize with the subsequent thinning of molybdenum layer is an important factor to be considered during the process flow of micromachined devices. The characterization of molybdenum oxide has revealed important findings regarding its behaviour and properties.

The phenomenon has been studied on two different samples. A thin layer (2000Å) of molybdenum has been deposited via magnetron sputtering using Evatec Clusterline on oxidized silicon substrates. One of these samples has been treated with a customized wet cleaning process and the second not. The presence and the kinetic of molybdenum oxide growth have been evaluated using ellipsometry (KLA ASET-F5x), surface resistivity (CDE ResMap Four Point Probe Resistivity Mapping System), XRD (Bruker J VX7300L) and TEM analysis. The thickness of molybdenum oxide measured by ellipsometry was found to be strongly dependent on surface treatments. After almost 60 days, molybdenum oxide thickness was 30 Å for untreated surface and 15 Å for the treated one [Fig 1]. The accuracy of the ellipsometric measurement has been validated by TEM analysis on tween samples, as shown in [Fig 2]. The oxidation process seems depending by a rather complex kinetics, showing a fast linear grown phase in the first ten/twenty days from deposition, depending by the surface treatment, followed by a slowdown of the growing rate. Assuming MoO₃ as the only species formed during the oxidation, from a stoichiometric evaluation, the consumption of molybdenum for a given layer of molybdenum oxide is approximately one third of the oxide thickness.

The growth kinetic of molybdenum oxide appears to be influenced by the chemical treatments of the metallic layer. The treatments with alkaline cleaning solutions have a passivation effect on the metal surface, influencing the total amount of oxide formed as well as the kinetic of oxidation. Sheet resistance measurements, slightly changing with time, shows an average higher resistivity of treated surface supporting this hypothesis [Fig. 3].

The results of this study provide valuable insights into the behaviour and properties of molybdenum oxide, which can be useful for various applications in the field of materials science. The observed non-uniformity in oxide thickness and the consumption of molybdenum due to oxide growth are critical considerations for the design and fabrication of MEMS structures. The influence of metallic layer thickness, morphology, and chemical treatments on the growth kinetics of molybdenum oxide highlights the importance of carefully control these parameters in MEMS fabrication.

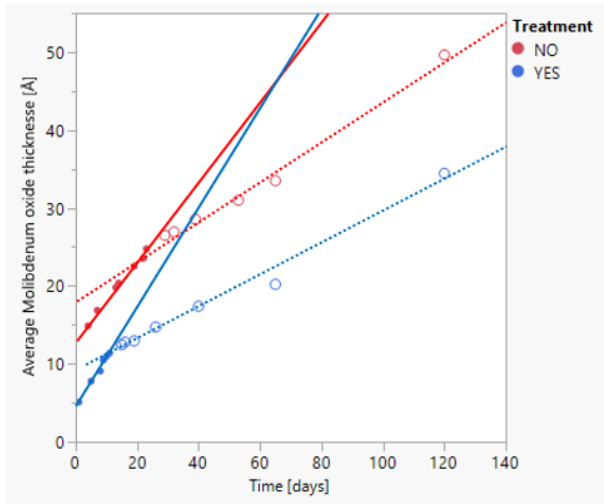


Figure 1. Ellipsometric measurements of molybdenum oxide along 120 days from deposition. Treated surface (blue) and not treated surface (red). Points refers to the average of 49 points measured on wafers. Solid and empty points refer to fast and slow kinetic phase respectively while solid and dotted lines are the respective linear trends.

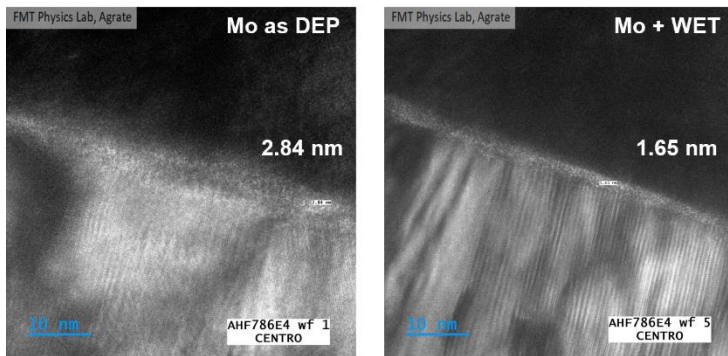


Figure 2. TEM images of molybdenum oxide after 60 days from deposition. Treated (Right) and not treated (left).

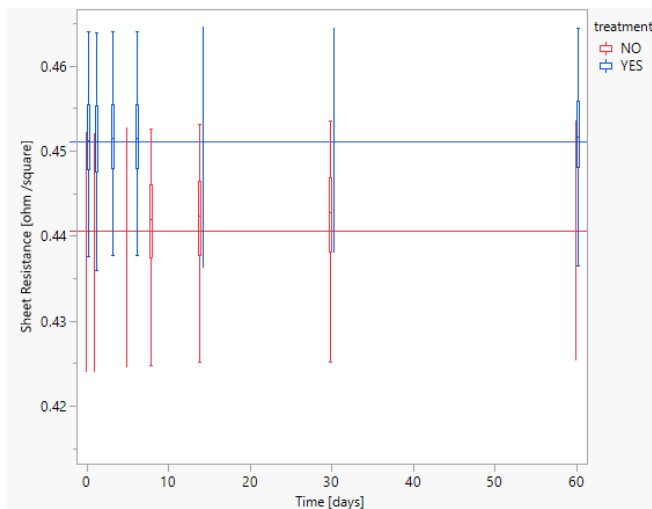


Figure 3. Surface resistivity measurements of molybdenum oxide along 60 days from deposition. Treated surface (blue) and not treated surface (red). Boxplot refers to the 49 points measured on wafers while solid lines refer to data average values.