Complementary electron microscopic-spectroscopic characterization of Ti and Cr adhesion layers

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Complementary electron microscopic-spectroscopic characterization of Ti and Cr adhesion layers

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The use of thin metal *adhesion layers* to improve adhesion has become standard in a wide range of applications and research areas, e.g. semiconductors, plasmonics, metamaterials, and 2D materials. The simplified model accepted in the micro and nanofabrication community involving an adhesion layer is shown in Fig. 1. In this model the adhesion layer and working over-layer are completely uniform and in perfect contact. However, the model does not take into account any interaction between layers and is considered valid for all thin film thicknesses. With the constant miniaturization that characterized micro and nanofabrication, the thicknesses of both adhesion layer and over-layer have reached tens of nanometers. Under these conditions it is not clear if such a simplified model is still valid: in particular there is little knowledge on the effect of the presence of an adhesion layer on the nanostructure of the over-layer. Furthermore, it is not clear whether the nanostructure is dependent on the type of adhesion layer used.

In this work, using complementary characterization techniques, we investigated how Cr and Ti adhesion layers influence the nanostructure of ultra-thin Au over-layers. For the morphology analysis, thin-film cross section samples have been prepared by focused ion beam (FIB). Analysis of SiO_2/Ti/Au and SiO_2/Cr/Au stacks showed the formation of a uniform Ti layer below Au, while a clear interface was not detected in the Cr/Au sample. STEM-EDX analysis of the same samples confirmed the different morphology, revealing complete Cr diffusion into Au as opposed to the Ti case (Fig. 2). Transmission Kikuchi Diffraction (TKD) [1] showed a change of grain size and orientation of the Au over-layer compared to pure Au for both adhesion layers. In particular, pure Au presented a bimodal grain size distribution, with [111] grains having an average size of 420 nm and [101] and [100] grains having a value of 50 nm (Fig. 3A). Ti/Au and Cr/Au samples had an Au average grain size of 50 nm and 35 nm (Fig. 3B and 3C), respectively. The surface of both adhesion layers is an efficient nucleation layer, decreasing Au diffusion and inhibiting island coalescence. The diffusion inhibition leads to the formation and growth of islands having the most stable growth direction and a homogeneous grain size distribution. For the case of Au, which has a face-centered cubic (FCC) unit cell symmetry, the most stable growth direction is [111]. Electron energy loss spectroscopy (EELS) showed the presence of oxygen bonded to both adhesion layers. For the Cr/Au sample, STEM-EELS linear scan showed three main signals: a Cr double edge, a SiO_2 O-K edge and a weak O-K edge of O bounded to Cr, visible only for a limited thickness below Au (Fig. 4A). Furthermore, the Cr edge presented a long diffusion tail into the Au layer, confirming EDX data. For this tail edge there was no presence of an O-K edge, indicating that Cr inside Au was in metallic form. X-ray photoelectron spectroscopy (XPS) analysis showed that the partial oxidation happened during film deposition for both adhesion layers. For the Cr/Au sample, to avoid possible oxygen migration from the oxygen-rich SiO_2 substrate, 2 nm of Cr and 20 nm of Au were deposited on amorphous Si_3N_4. After Ar ion milling of 20 nm of Au, Cr 2p and O 1s signals started to appear at the same depth (Fig. 4B and 4C, respectively). Micro 4-point probe (µ4PP) measurements were performed to investigate possible impacts of the Au over-layer nanostructure change on the macroscopic properties e.g. electrical resistivity. A µ4PP SEM module (from Capres A/s) connected to a Kleindiek Nanotechnik MM3A-EM micromanipulator mounted inside a FEI Nova 600 SEM was used for the analysis. With this system, sheet resistance (R) was measured for the three samples investigated by TKD: 20-nm-pure Au, 2-nm-Ti and 20-nm-Au, 2-nm-Cr and 20-nm-Au. The measurements presented an electrical conductivity increase for Ti/Au and decrease for Cr/Au stacks compared to pure Au, attributed to film parallel resistor behavior [2] and Cr/Au alloy formation, respectively.

Based on these results, a revised adhesion layer model is proposed, which takes into account film morphology, texture, elemental diffusion properties and chemical composition of a general adhesion layer/over-layer system. A fully descriptive model is essential in thin-film engineering, as it enables researchers to predict and choose the adhesion layer that fits better their needs.

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[2] Y. Y. Chen, J. Y. Juang, Meas. Sci. Technol. 27 (7) (2016), 74006.

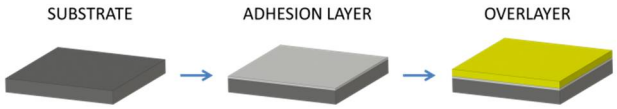

Figure 1. Simplified model of substrate-adhesion layer-overlayer system.

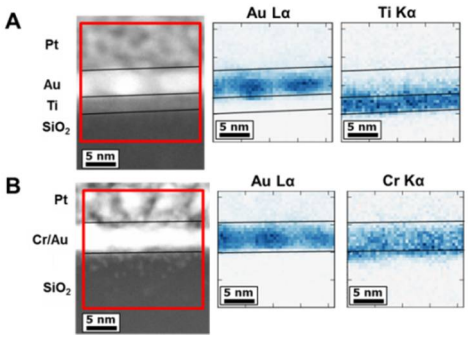

Figure 2. STEM-EDX map of: (A) 2-nm-Ti and 2-nm-Au, (B) 2-nm-Cr and 2-nm-Au samples.

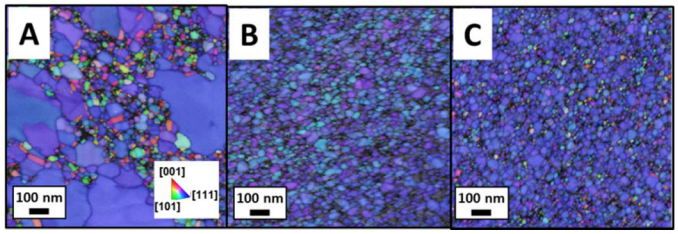

Figure 3. TKD IPFZ maps of 20-nm-Au (A), 2-nm-Ti and 20-nm-Au (B), 2-nm-Cr and 20-nm-Au (C) samples.

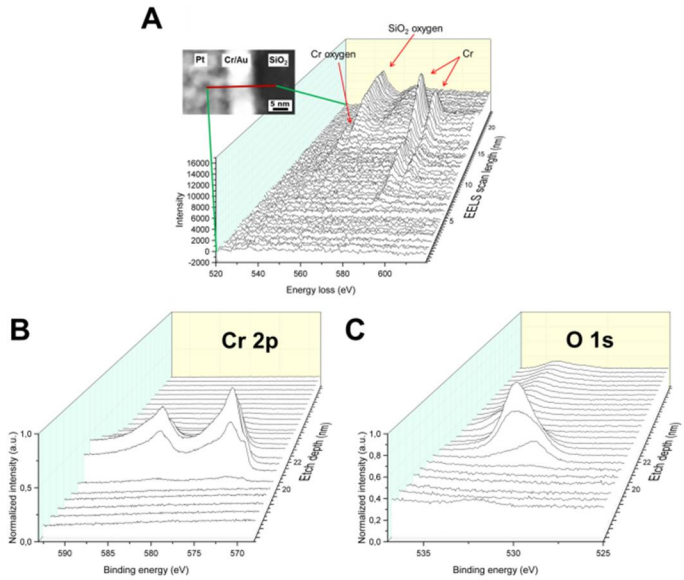

Figure 4. STEM-EELS linear scan of 2-nm-Cr and 2-nm-Au (A). Cr 2p (B) and O 1s (C) XPS peaks of 2-nm-Ti and 20-nm-Au sample XPS depth profile on Si_3N_4 substrate.