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Investigation with complementary characterization methods of adhesion layer effect on nanostructure of gold ultra-thin films

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Adhesion of metal thin-films to dielectric or semiconductor substrates is important in a wide range of applications and research areas, e.g. semiconductors, plasmonics, metamaterials, nanopores, graphene, MoS_2 and other 2D materials. Many important metals used for such applications (e.g. Cu, Ag, Au) are strongly affected by poor mechanical adhesion to the substrate, leading to performance reducing phenomena like delamination and time dependent device performances deterioration. In order to minimize these problems the so-called *adhesion layers* have been introduced. The most important adhesion layer metals are Ti and Cr. Both in research and industry the thicknesses of adhesion layers and functional metal thin-films (over-layers) have reached comparable values (tens of nanometers) and therefore the adhesion layer starts to influence on the thin-film structure. To our knowledge there are no reports on adhesion layer effects on the nanostructure of metallic ultra-thin over-layers. Nanostructure of thin-films is an important microscopic property as it is directly connected to macroscopic physical properties, i.e. electrical conductivity. Metal thin-films are usually polycrystalline, and their electrical conductivity shown a significant reduction compared to bulk values, which has been mainly attributed to surface and grain boundary scattering [1]. Comprehension of the adhesion layer dependence of thin-film nanostructure could therefore have an important impact on fabrication of nanodevices with superior electrical performances.

Using complementary characterization methods, we investigate how Ti adhesion layers influence the nanostructure of ultra-thin Au over-layers. We present two samples: one with 20 nm Au layer and another with 10 nm Ti adhesion layer and 20 nm Au over-layer. Thin-films are deposited by e-beam evaporation at room temperature on 5 nm SiN TEM grids. Bright and dark field micrographs and 20 µm selected–area diffraction patterns (SADP) have been acquired using an FEI Tecnai T20 G2 TEM at 200 kV to investigate thin-film grain size, elemental composition, and crystal orientation. Diffraction patterns have been indexed with JEMS software. Bright field micrographs of the two samples are shown in Fig. 1A (20 nm Au) and 1D (10 nm Ti + 20 nm Au). No appreciable difference in nanostructure is visible from their comparison. For the 20 nm Au sample, a diffraction pattern is shown in Fig. 1B. Five crystallographic planes have been identified: (111), (200), (220), (311) and (222). A dark field micrograph using the (111) and (200) diffraction spots is shown in Fig. 1C. A statistical analysis performed with Image J program shows an average grain size of 15 nm (inset in Fig. 1C). The diffraction pattern of the 10 nm Ti and 20 nm Au sample is shown in Fig. 1E. We observe a more complicated diffraction pattern structure: several diffraction rings of Ti and Au are superimposed, in particular Au (111)/ Ti (002) and Au (220)/ Ti (110) strong reflections. For the indexing, hexagonal-close packed structure of Ti has been selected. In the correspondent dark field micrograph for diffraction spots Au (111)/ Ti (002) a simultaneous scattering of the two elements is present and grains with sizes ranging from 5 to 40 nm are visible (Fig. 1F). Bigger grains can be due to coalescence, but it is not possible to distinguish between single grains of Ti and Au due to the finite size of objective aperture, that does not permit selection of superimposed or very close diffraction rings. Energy dispersive x-ray spectroscopy (EDX) of the samples confirms of their elemental composition (Fig. 2).

Further studies of Au grain size and orientation mapping will be performed by the use of transmission Kikuchi diffraction (TKD) [2]. In-situ annealing in the TEM/SEM will allow gaining information on nanostructure change of thin films under high-temperature working conditions present in MEMS/NEMS and FETs.

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[2] R. R. Keller, R. H. Geis, Journal of Microscopy 245 (3) (2012), 245-251.

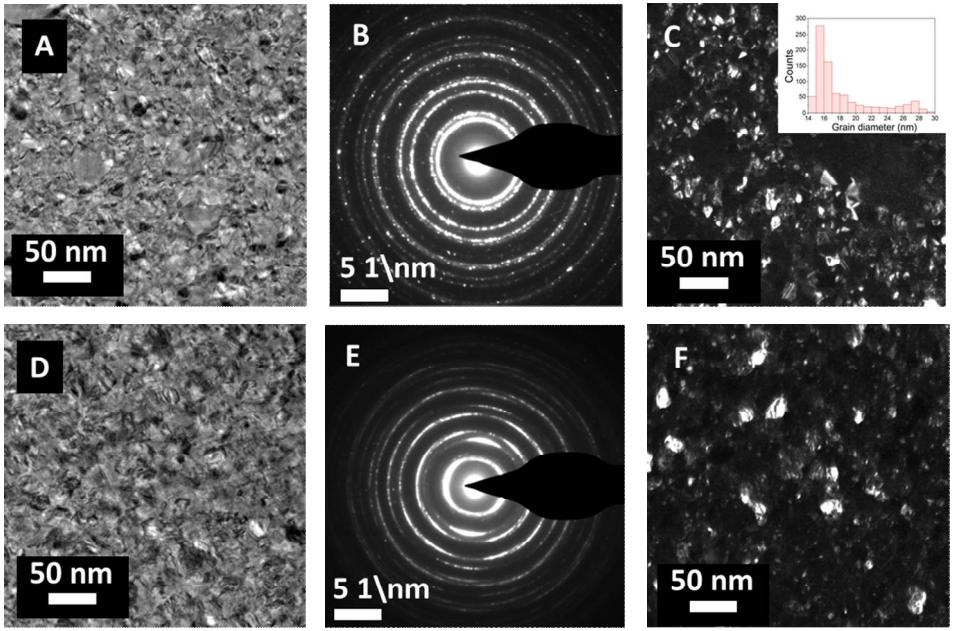

Figure 1. Diffraction patterns, bright and dark field micrographs of analyzed samples. (A)-(C) 20 nm Au sample. (D)-(F) 10 nm Ti + 20 nm Au sample.

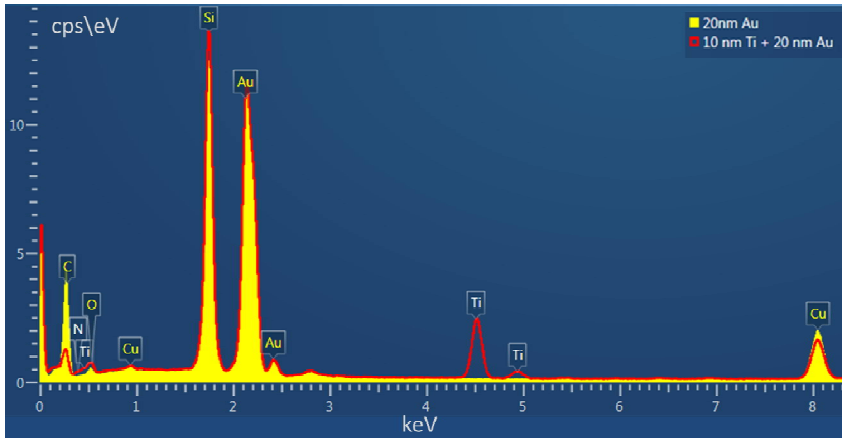

Figure 2. EDX spectra comparison of the analyzed samples.