DEVELOPMENT AND APPLICATION OF AN IN SITU-SEM NANOINDENTER COUPLED WITH ELECTRICAL MEASUREMENTS

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The increasing demand for multifunctionality has become a recurring challenge for a wide panel of application fields such as microelectronics and structural applications. In that purpose, a multifunctional characterisation set-up is being developed at SIMaP lab, mainly based on electrical and mechanical coupling. The heart of this device is an in situ-SEM nanoindenter coupled with an electrical measurement apparatus. This device aims at improving the understanding of mechanical behaviors such as thin film delamination or oxide layer fracture. In the present communication, instrumental developments and experimental results will be detailed.

Different critical aspects of the experimental procedure will be discussed: 1/ In order to sense fast mechanical events such as oxide fracture or dislocations bursts, a highly sensitive electrical instrument is required. The apparatus used in this study [1] was able to measure large resistance changes over ten decades and to work at high frequency (kHz). 2/ The choice of the conductive tip was also crucial. Both standard boron-doped diamond (BDD) and tungsten carbide (WC) tips have been tested in this study. 3/ Different local environments have also been assessed: room atmosphere, dried-atmosphere, intermediate and high vacuum. Characterisations have then been performed on model specimens: noble metal (gold) or natively oxidised metals (copper and aluminum), either as bulk single crystals or as polycrystalline thin films. Electrical resistance was measured continuously during indentation under different applied voltages.

This benchmark approach clearly emphasized the contrasted behaviors of the different materials (Figure 1). Possible scenarios are proposed to relate the obtained results to the microstructure, the oxide layer and electrochemical processes. Changes in electronic transport regimes, local resistivity estimations and real-time monitoring of mechanical contact area are also investigated.

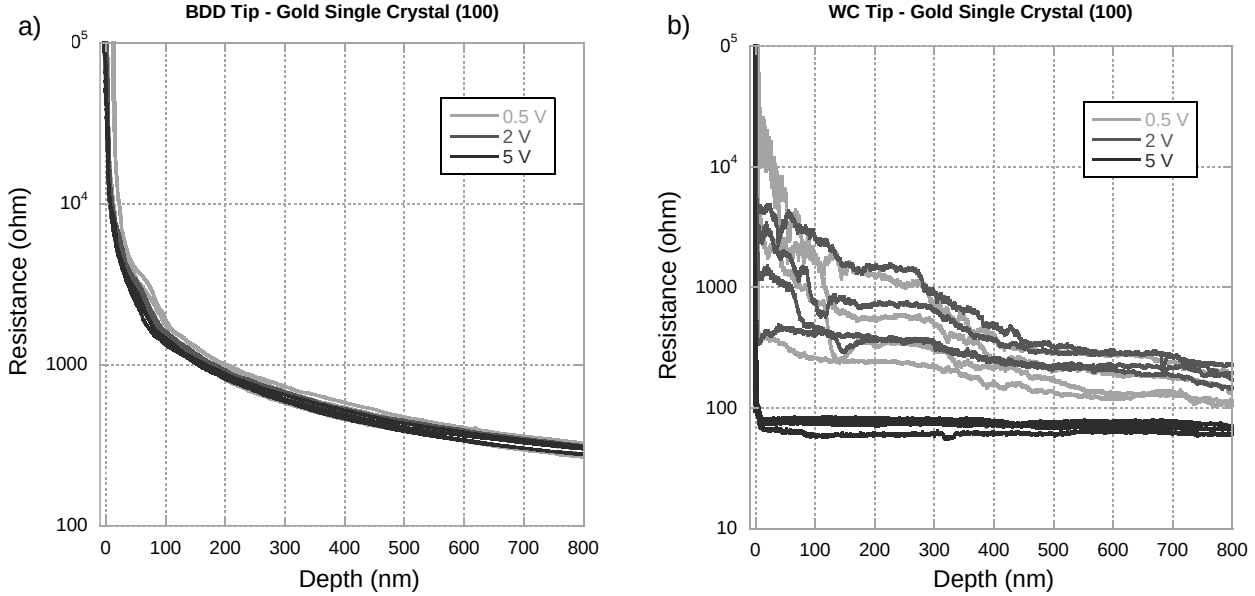

Figure 1 – Resistance versus depth curves for different sample polarization a) BDD tip and Au sample b) WC tip and Au sample.

[1] F. Houze et al., Imaging the local electrical properties of metal surfaces by atomic force microscopy with conducting probes, Appl. Phys. Lett., 69, pages 1975-1977, 1996