Development and application of an in situ SEM nanoindenter coupled with electrical measurements

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Jury :
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Abstract: The increasing demand for multifunctional materials has become a recurrent challenge for a wide panel of application fields such as microelectronics and structural applications. Within the frame of this project, a multifunctional characterisation set-up has been developed at SIMaP lab, mainly based on the electrical / mechanical coupling. The heart of this device is an in situ FEG-SEM (Field Emission Gun Scanning Electron Microscope) nanoindenter coupled with an electrical measurement apparatus. This work has threefold objectives: (1) The investigation of mechanical behavior of small scale systems, (2) The input of electrical data to the quantitative analysis of mechanical behavior during indentation, in particular to obtain a better estimation of the contact area (3) The local study of electronic properties of thin film stacks. SEM integration of the device has been validated and indent positioning with a precision better than 100 nm is successfully obtained. This performance allows the studies of mechanical properties at submicrometric length scale, with a high throughput allowing statistical measurements. Various bulk composite materials have been characterized as well as submicrometric gold islands on sapphire. In the latter case, despite the stochastic nature of their mechanical behavior, a deterministic law has been extracted. 3D-BCDI (Bragg Coherent Diffraction Imaging) experiments have been performed on a few islands at synchrotron facility to investigate the crystal state before and after mechanical loading. These experiments reveal initial dislocation nucleation prior to large deformation bursts. In parallel to this study, electrical measurements have been performed during indentation on various cases. Resistive-nanoindentations have been performed on noble metals (Au) and natively oxidized metals (Cu, Al), either as bulk single crystals or as polycrystalline thin films. Qualitative results emphasize the importance of the oxide layer on the electrical response. In the presence of an oxide layer, strong electrochemical reactions seem to occur at the tip-to-sample interface. When no oxide is involved, the measured resistance can be fully described by an analytical model and the computed electrical contact area is successfully validated with residual areas measurements. Finally, capacitive measurements have been performed on MOS structures with various oxide thicknesses. Experimental results have been well described by analytical

modelling, which paves the way for quantitative local dielectric permittivity measurements under mechanical loading.