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Effect of Induced Stimuli on the Leakage Current of Operative Oxide-based Devices inside a TEM

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Nanoelectronics devices are guided by their electrical performance under operative conditions. Therefore, a detailed understanding of the correlation between the electrical properties and the material structure at the nano and sub-nano levels is needed. In this way, microelectromechanical systems (MEMS) for in situ transmission electron microscopes (TEM) offer the possibility to perform multi-stimuli experiments comparable to real working conditions [1]. However, to this date, biasing of *e.g.*, Metal-Insulator-Metal (MIM) devices inside a TEM using MEMS-based chips has not been fully achieved due to its complex sample preparation and the generation of parasitic stray leakage paths. Here, a focused ion beam (FIB)-based preparation of TEM lamellas free of short-circuits is shown. Additionally, a study of the effect of induced stimuli on the device's leakage current during working conditions is also demonstrated.

Various examples of in situ TEM biasing using MEMS chips have been reported [2]–[4]. Nevertheless, most of the current levels shown in the literature are too high for a TEM lamella-sized device, mainly due to the formation of undesired short-circuits along with the chip during sample contacting. Recently, a novel method was in-house established showing low leakage currents compared with a short-circuited sample. The current density values obtained with this novel approach are equivalent to the ones obtained to its corresponding millimeter-sized devices. In this fashion, the bottleneck of having operando electronic devices for in situ TEM has been overcome. Therefore, a direct correlation of the electrical properties and structural changes at the micro and/or sub-nanometre level is now feasible. For instance, here, we present the effect of induced stimuli in the current levels of contacted and operative TEM lamellas.

First, confirmation of operando conditions by comparing the current density (J) versus the electric field (E) between millimeter-sized devices and TEM lamellas extracted from the same device are analyzed. In this regard, four different oxide devices (RRAMs, Varactors) with their corresponding TEM samples were compared. Second, the effect of induced stimuli to the sample during biasing conditions can be online tracked via its current readouts. Thus, the monitoring of current in a gas-biasing experiment inside the TEM was also performed, finding a suppression of leakage current due to oxidation of the oxide thin films. Moreover, the effect of electron beam irradiation on the electrical properties of oxide devices is also considered.

Our successful FIB-based sample preparation routine for operando TEM biasing studies using MEMS-based chips allows the investigation of nanoelectronics devices under multi-stimuli environments (heating, biasing, gas). A direct comparison between millimeter-sized samples and corresponding TEM lamellas is obtained via the I-V curves and used to establish an appropriate contacting of TEM lamellas for in situ biasing. In the same manner, we monitor the electrical properties simultaneously with induced stimuli such as gas-biasing and electron beam irradiation-biasing experiments. The current-driven mechanisms (currently under investigation) can be associated with the corresponding microstructural features *e.g.*, local defects, oxygen vacancies, antisite defects, formation of domain walls, etc.

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