Characterization of functional materials by in situ SEM electrical nanoindentation

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Abstract: his PhD thesis belongs to the trend toward the development of innovative materials for multifunctional applications (in micro/nanotechnology, metallurgy, ...). Indeed, the "Materials" specifications of these developments require more and more the combination of various properties (mechanical, electrical, dielectric, ...) sometimes antinomic, all at submicronic scales. The characterization of these complex systems therefore requires techniques adapted to these constraints. The work carried out during this PhD is based on the development, improvement and application of an innovative multifunctional characterization technique: the nanoindentation coupled to electrical measurements and integrated in a SEM. The device used in this study was developed in SIMaP laboratory. This instrument is a nanoindenter, initially dedicated to mechanical tests, which has been functionalized to perform simultaneously electrical and mechanical measurements. Moreover, the present device can be integrated into SEM to ensure the positioning of the indenter with a resolution of the order of a hundred nanometers, as well as to visualize physical events in real time. The aim of this work is to apply this innovative technique to three systems of industrial interest to study their local electrical and mechanical properties. The first studied material is a multiphase metallic alloy composed of silver, copper and palladium (AgPdCu). Thanks to the electrical measurements associated with nanoindentation tests, a complete methodology (partly based on the current-voltage characteristics) has been developed to continuously monitor the evolution of the contact area during penetration of the tip in the material. In addition, SEM allowed to position the indentor at the center of the micrometric phases, thus avoiding time-consuming statistical experiments. Using this methodology, the elastic modulus and hardness of the individual phases were successfully determined. The second investigated system is a piezoelectric structure consisting of aluminum nitride (AlN) islands deposited on silicon pillars. The effective piezoelectric coefficient was measured using a flat punch inducing a perfectly elastic mechanical response of the system. The extracted coefficient is in good agreement with the values reported in literature, which shows the effectiveness of the electrical nanoindentation in situ SEM technique for this type of measurement. The same material was indented using a Berkovich tip inducing plasticity, showing a much different response most likely due to the structural defects

injected into the material. The third characterized system is a stack of thin films including a silicon nitride film (Si3N4) deposited on a metallic layer (AlSiCu). Leakage currents through the dielectric material have been measured and correlated to its mechanical degradation. Coupling of the nanoindenter device with SEM allowed to in situ visualize the cracking of the film in real time. The effect of a brittle or ductile underlayer on the mechanical response of the system was also studied with the support of numerical simulations conducted by finite element method (FEM). Results showed that in the presence of a ductile underlayer, the predominant mechanism is the cracking of the brittle film, while in its absence, the predominant mechanism is the plasticity of the brittle film. To conclude, the present study showed that the electrical nanoindentation in situ SEM technique is a perfectly suited tool to perform high sensitivity local electrical and mechanical measurements at submicron scales. This technique can now be transposed to other systems whose electrical properties depend on mechanical stimuli and vice versa.