

### **Mechanical characterization of battery nanomaterials**

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Mechanical degradation of electrodes is a key factor in the loss of capacity observed in battery materials during extended cycling. This degradation mechanism has its origins in mechanical stresses, cracking, and / or fracture, which are induced within the battery electrode, due to the volume, compositional, or phase changes that occur during the intercalation of inorganic ions (such as  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ , etc.) into the host crystal. An electrochemically correlated understanding of intercalation-induced changes in electrode Young's modulus, volume / geometry, and fracture strength is critical to alleviating these mechanical degradation mechanisms and to thereby, increase the cycle-life of batteries. This effort will present an AFM-based, single-nanowire battery diagnostics platform to characterize these performance metrics in electrode nanomaterials. The on-chip platform is created by assembling doubly-clamped, single nanowire electrode beams across spatially separated current collectors, which are made of gold nanostructures defined using electron beam lithography and metal lift-off. The single nanowire electrode beams are assembled on to the current collectors from their colloidal suspensions in alcohol-based solvents, using dielectrophoretic nanomanipulation techniques. The post-assembly nanowire structures are further clamped on to the gold electrodes using electron-beam induced deposition of platinum metal. Furthermore, the state of charge of these single-particle electrodes is controlled by intercalating inorganic ions inside a glovebox. Next, contact-mode, atomic-force microscopy (AFM) based three-point bending tests are performed on these nanoelectrode beams to extract their Young's modulus and fracture strength. In this technique, a pre-calibrated AFM tip is used to push on the suspended nanobeams and the deflection of the nanowire is monitored as function of the controlled flexural force, which is applied by the AFM tip. Analytical models are then implemented to obtain the Young's modulus and fracture strength values of the nanobeams from this experimental data. These measurements at the single-particle level provide accurate insights into the intrinsic performance of the electrode material system and represent an improvement from past measurements involving either microscopic aggregates of particles, which include influences from inter-particle interfaces, or thin film / bulk-scale electrodes, which include influences arising from grain-boundaries and crystal defects. This effort will present results from this experimental approach involving one-dimensional battery material systems. These results point to the relevance of material parameters such as crystallinity, geometry / size of ionic intercalation sites, growth orientation of the crystal, exposed facets, and surface layers. The presented experimental approach is anticipated to be a useful tool for studying and alleviating mechanical degradation in next-generation battery electrode material systems.