Accurate mechanical phase mapping in heterogeneous materials combining high-speed nanoindentation with a novel Machine learning-driven pile-up error correction

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Abstract

Accurate mapping of mechanical properties across extensive areas in heterogeneous materials is essential for understanding phase-specific contributions to strength and hardness. High-Speed Nanoindentation Mapping (HSNM) enables their x-y spatial mapping through a fast and dense grid of indents. However, truthful measurements are complicated by *pile-up*, the plastic displacement of material laterally and vertically around an indent, often causing hardness and modulus overestimation, especially in materials with varying phase compliance. Traditional correction methods rely on time-consuming, localized Atomic Force Microscopy (AFM) measurements, which are impractical for large-area mapping.

This study presents a fast, generalized, and semi-automated method for evaluating and correcting pile-up effects in high-speed nanoindentation mapping (HSNM) leveraging the induced measurable changes in surface roughness (Sa), which are quantifiable through fast optical profilometry. By correlating these changes in roughness with the pile-up height measured via atomic force microscopy (AFM), universal calibration functions were derived and validated for a wide range of bulk materials and thin films, with additional verification provided through Finite Element Modeling (FEM). The method was demonstrated on 1-micrometer thick gold (Au), copper (Cu), and titanium (Ti) coatings on silicon substrates, as well as mechanically polished bulk titanium, aluminum, copper alloys, and bulk metallic glass samples. High-speed nanoindentation arrays were produced at different indentation depths (300, 500, 600, and 1000 nanometers), using a 10-times spacing rule to generate varying levels of pile-up. In bulk materials, a unique linear correlation was observed between changes in surface roughness and pile-up height, while coatings exhibited distinct slopes with a clear linear correlation maintained. This method offers a robust basis for quickly estimating pile-up height and correcting hardness and elastic modulus values, overcoming limitations posed by conventional AFM acquisition and expanding the applicability through the integration of machine learning for selective pile-up correction over large areas.

Applied to a benchmark Co-Cr-W hard metal alloy, the method uses unsupervised clustering to identify piling-up phases in the cobalt matrix while excluding the hard carbides. This

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approach reduced the hardness and modulus errors by up to 7%, uniquely enabling accurate phase-specific property mapping in high-speed nanoindentation advancing the mechanical microscopy frontier.