A SIMPLE METHOD FOR PILE-UP CORRECTION BY HIGH-SPEED NANOINDENTATION COMBINED WITH OPTICAL PROFILOMETRY

Razvan Lucian Natea, Engineering Department, "Roma TRE" University, Via della Vasca Navale 79, Rome 00146, Italy
raz.natea@stud.uniroma3.it
Edoardo Rossi, Engineering Department, "Roma TRE" University, Via della Vasca Navale 79, Rome 00146, Italy
Saqib Rashid, Engineering Department, "Roma TRE" University, Via della Vasca Navale 79, Rome 00146, Italy
Marco Sebastiani*, Engineering Department, "Roma TRE" University, Via della Vasca Navale 79, Rome 00146, Italy

Key Words: Nanoindentation; Pile-up; Optical profilometry; AFM;

Theoretical and experimental studies have widely investigated the influences of pile-up on hardness and modulus calculation by nanoindentation [1]. In those cases where this phenomenon is relevant, the most effective solution is to perform time-consuming direct measurements of the actual contact area on individual indentation marks (e.g., by Atomic Force Microscopy).

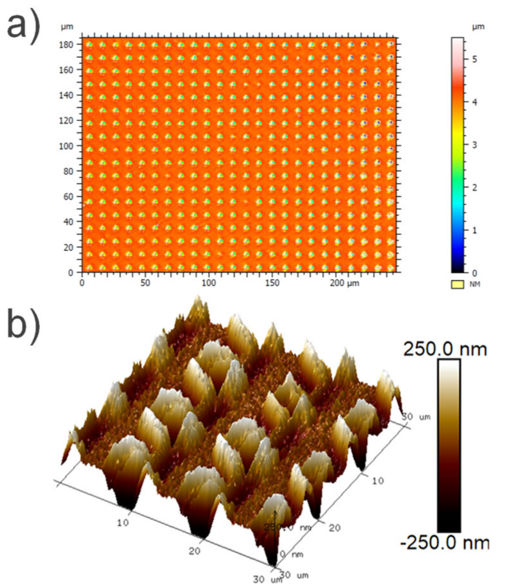

Figure 1 – a) representative profilometry acquisition for a 50x objective on a 1000 nm depth indentation array (1 μm Au film) and b) corresponding AFM scan for measurements' calibration.

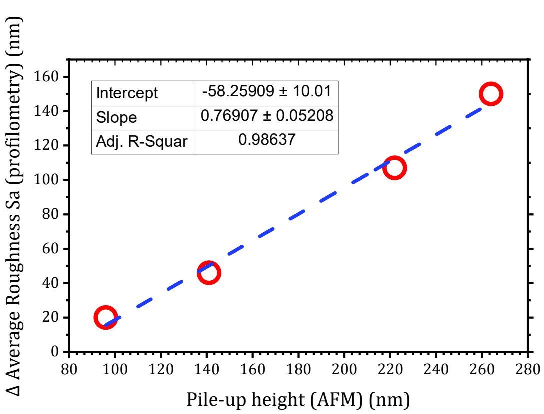

Figure 2 –Correlation between profilometry evaluated roughness (Sa) change in the Au indented film and AFM measured pile-up height for a 50X objective.

This work presents an alternative fast and straightforward method for evaluating and correcting pile-up effects in nanoindentation experiments.

The main idea is to use high-speed nanoindentation to produce a large array of indents which generates a measurable change in the surface roughness quantifiable by fast optical profilometry analysis. It is demonstrated that the change in surface roughness (Sa) caused by the array of indents is strongly and uniquely correlated to the pile-up height.

To validate this idea, several materials were investigated: a one μm thick gold (Au) film on Silicon substrate, fine mechanically polished aluminium alloy and copper alloy samples. In all cases, high-speed nanoindentation arrays were realized at different indentation depths (300 nm, 500 nm, 600nm and 1000 nm), following a $1/10th$ spacing rule, to induce distinct pile-up levels.

Additional experiments have also been performed on a fused quartz reference sample to further elucidate and validate the proposed methodology in those cases where sink-in effects are dominant in the material's indentation behaviour. The actual pile-up height was measured by atomic force microscopy (Figure 1b). At the same time, the surface roughness (inside and outside the array of indents) was analyzed by confocal optical profilometry (Figure 1a) and roughness changes related to the evaluated AFM pile-up height. The procedure has been repeated for several magnification levels: 20X, 50X, 100X, and 150X objectives.

The results show a linear correlation between the change in surface roughness and the pile-up height (Figure 2). Finding are extended to all the investigated materials and adopted optical objectives.

This work, therefore, demonstrates that the proposed alternative methodology for pile-up assessment is stable over different materials. The obtained correlation functions could provide a direct basis, being provided the mapping of a family of calibration coefficients over different material properties and magnifications, to estimate pile-up height and consequently perform correction of the hardness and elastic modulus in a fast and reliable way, overcoming limitations imposed by AFM acquisitions.

[1] McElhaney, K. W., Vlassak, J. J. & Nix, W. D. J. Mater. Res. 13, 1300–1306 (1998).