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Single crystal plasticity parameters identification from residual imprint topography after nano-indentation

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Abstract — Fundamental deformation mechanisms of FCC materials under indentation have been probed at the grain scale. Experimental tests have been conducted on large-grained annealed and cold-worked polycrystalline nickel samples with a Berkovich indenter. Indentation axes have been chosen to be close to the three main crystallographic directions [001], [101] and [111]. Pile-ups and slip traces have been revealed around the residual imprints by analyzing topographic measurements obtained by atomic force microscopy. It is shown that the indenter orientation in each indentation plane drives pile-ups and slip traces which in turn contain precious information about the crystallographic orientation and the hardening state of the studied grain. Imprint topographies after pile-up formation therefore carry information that one can exploit to assess some intrinsic material properties at the grain scale. A 3D finite element modeling of the nano-indentation test at the grain scale has been developed, making use of crystal plasticity constitutive laws. Six different virtual materials having the same macroscopic behavior have been built. The simulation results show a good agreement with experimental tests and also a great pile-up sensitivity to interaction matrix components. These results pave the way to the interaction matrix identification using an inverse finite element method.
Keywords — AFM, identification, single crystal plasticity, nano-indentation

Introduction Whether based on continuum mechanics or dislocation dynamics, crystal plasticity models involve generally a large number of material parameters, making the description of the phenomena very complete [1]. In this way, the critical resolved shear stresses for the different slip or twinning systems (CRSS), hardening parameters accounting for dislocation interaction, dislocation annihilation or nucleation terms can lead to models making use of more than 20 parameters. The extraction of each one of them from experimental mechanical data by inverse method is thus challenging. In this aim, assessing the uniqueness and the sensitivity of the identified set of parameters is crucial.

We investigate herein the information contained in the residual topography obtained after an indentation test. As suggested by Zambaldi *et al.* [2], this topography can be used as a fingerprint of the underlying crystal deformation processes. As pile-up distribution around an indentation imprint performed on a single crystal strongly depends on the indented crystal orientation, it may serve the identification of single-crystal plasticity parameters.

Experimental The experimental part makes use of three polycrystalline nickel samples. The first nickel sample (a) has very small grains (average grain size about 0.37μm) and was obtained by electro-deposition with a thickness of 300μm. The second one (b) is a 99.9% pure nickel sample (Goodfellow society) with large grains (average grain size about 140μm) and was obtained after 3 h annealing at 900^o C of a cast ingot. The last one (c) is obtained by work-hardening (30% in traction) the annealed nickel sample (average grain size of about 168μm). Samples have been indented at room temperature with a Berkovich diamond tip, so as to always obtain an indentation depth of about 900 nm. Indentations have been performed in three different crystallographic planes close to the $\{001\}$, $\{101\}$ and $\{111\}$ family of lattice planes, with varying indenter's orientations in the lattice plane. All indentation imprints have been observed with an atomic force microscope to reveal the pile-up distribution and sizes. These results particularly demonstrate a strong dependence of the imprint to the indenter's orientation. It is also recovered that the residual deformation amplitude strongly depends on the hardening rate.

FEM simulations Three dimensional numerical simulations of the nano-indentation test have been performed using the finite element code Zebulon [3], using elastic parameters from the literature, plausible viscoplastic parameters and different sets of micro-plastic parameters obtained from a tensile stress-strain curve, and assuming a particular term of the interaction matrix is dominant. These FEM simulations yield numerical topographies which are compared to the experimental ones (See Figure 1 for the plane $\{001\}$). The comparison is qualitatively good, and these simulations additionally yield indications of the sensitivity of the topography to the various terms in the interaction matrix.

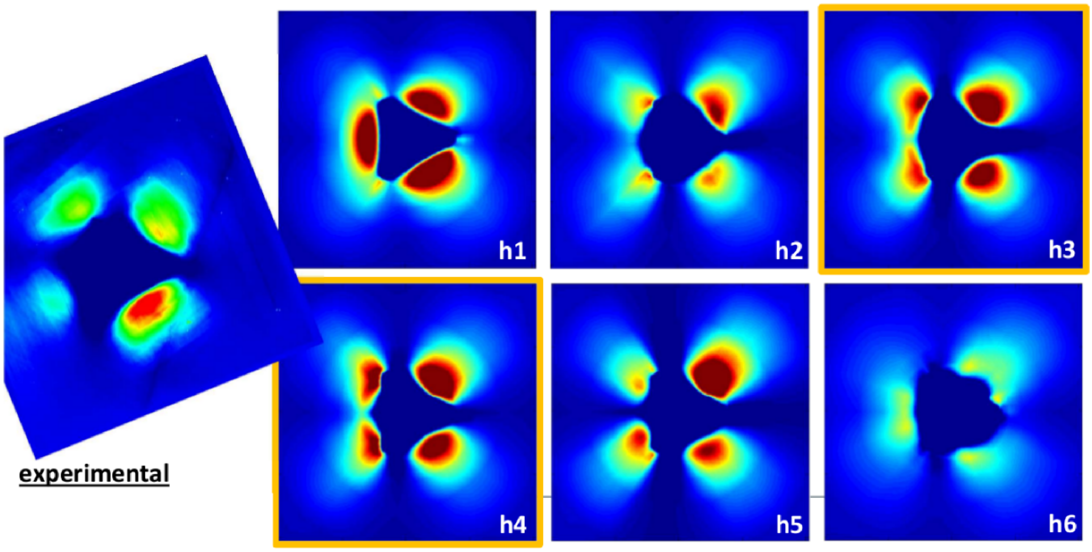

Figure 1: Experimental and various computed residual topographies (plane $\{001\}$).

Conclusion Pile-up distribution experimentally observed around residual imprint of indentation is strongly related to the indented crystallographic orientation and to the Berkovich indenter orientation in a given indented plane. Moreover, hardening rate appears to play a crucial role on the pile-up dimensions. A numerical modeling of the nano-indentation test has been implemented in a finite element code. The parameters of the single crystal plasticity material model previously identified have been introduced. Numerical simulation results are in a good agreement with the experimental observations: pile-ups are distributed in the same way. Moreover, pile-up heights and widths share the same order of magnitude. Therefore, an inverse finite element method from a Berkovich nano-indentation experiment (residual topography, indentation curve), appears to be a possible way to extract some of the material's constitutive parameters involved in the Meric-Cailletaud's model. Above all, there may be enough information in measured topographies to estimate some components of the interaction matrix.

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