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CROSS-SECTIONAL MICROSTRUCTURE AND STRESS DISTRIBUTIONS IN THIN FILMS DURING INDENTATION REVEALED BY X-RAY NANODIFFRACTION

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ABSTRACT

In this contribution, results from cross-sectional X-ray nanodiffraction experiments on thin films will be presented. The films were analysed at synchrotron facilities ESRF in Grenoble and DESY in Hamburg ex-situ after or in-situ during indentation using X-ray beams with a diameter down to 30nm. The results reveal complex microstructure and stress changes, which magnitude correlates with the observed cross-sectional fracture modes in the area of the indentation imprint.

Keywords: thin film, indentation, synchrotron X-ray nanodiffraction.

INTRODUCTION

Load-displacement curves measured during indentation experiments on hard thin films depend on non-homogeneous intrinsic film microstructure and residual stress gradients as well as on their changes during indenter penetration into the material. To date, microstructural changes and local stress concentrations resulting in plastic deformation and fracture were quantified mainly by numerical models, which suffer from poor knowledge of size dependent material properties and the unknown intrinsic gradients. Additionally, indentation-induced microstructural changes have been extensively studied by ex-situ and in-situ transmission electron microscopy (TEM). TEM studies do not provide however magnitudes of internal stress distributions and microstructural changes occurring in thin film volume under and around the contact area during the indenter tip penetration.

In this contribution, results from ex-situ and in-situ synchrotron X-ray nanodiffraction experiments, which were used to resolve simultaneously microstructural changes and stress distributions in various hard thin films during or after indentation, will be presented. Additionally, complementary finite element (FE) models as well as scanning and transmission electron microscopy (SEM and TEM) will be presented.

RESULTS AND CONCLUSIONS

In Figure 1(a), a schematic setup of an in-situ X-ray nanodiffration experiment (Keckes, 2012) is presented. This unique setup installed at DESY in Hamburg (Zeilinger, 2016) has been used to characterize microstructural changes and stress concentration in TiN thin film

during indentation. Similar studies were performed also at ESRF in Grenoble and will be presented in the contribution.

Figure 1(b) shows an example of a cross-sectional residual stress distribution in Cr sublayers of a multi-layered CrN/Cr thin film are presented (Stefenelli, 2015). The data were recorded after the indentation ex-situ.

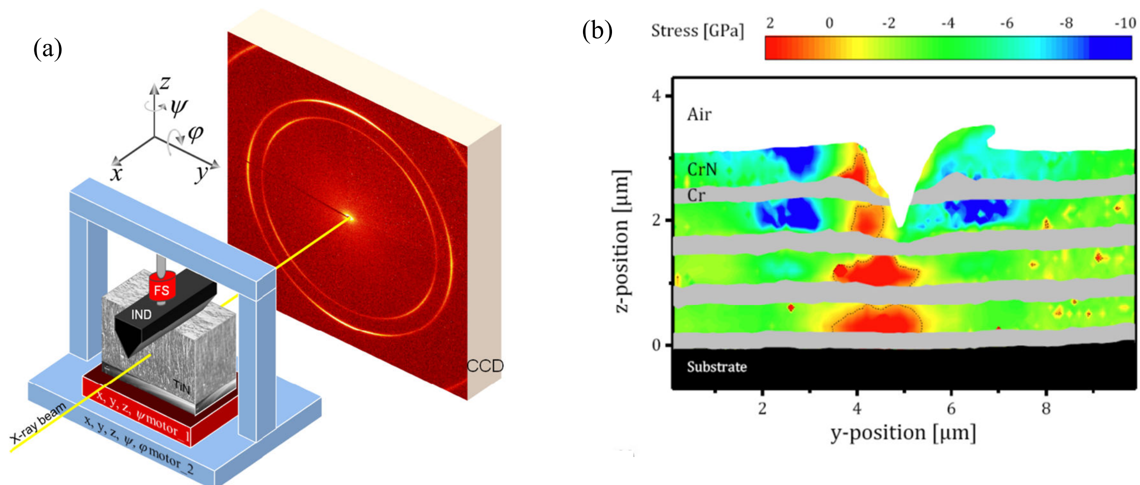

Fig. 1 - A schematic setup of an in-situ X-ray nanodiffraction experiment on a TiN thin film (a), which is indented using a wedge indenter (IND) (Zeilinger, 2016). During the indentation, the indenter platform with the sample is moved in the X-ray beam and the diffraction data are collected using a 2D detector (CCD). In (b), a cross-sectional distribution of in-plane residual stresses in CrN sublayers of CrN/Cr thin film recorded after an indentation indecates high compressive and tensile stress concentrations (Stefenelli, 2015).

In this contribution, results from ex-situ and in-situ experiments on various multilayered and monolithic thin films will be shown and the correlations to observed fracture modes will be discussed. The experimental data will be correlated with FEM models as well as complementary TEM results.

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