MS28 P01

Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction P. Goudeau[a], G. Geandier[a], P.-O. Renault[a], N. Tamura[b], C. Coupeau[a], F. Foucher[a], [a]*LMP, University of Poitiers, France.* [b]*ALS – LBNL, Berkeley, USA.* E-mail: pgoudeau@univ-poitiers.fr

Keywords: metallic coatings, mechanical stresses, buckling

The understanding of the mechanical properties of coated materials is a key factor in a number of technological applications. In particular, the delamination of compressed thin films is one of most limiting factor for the structural performance of the material and presents various interesting problems in physics and mechanics.

The delamination patterns result from relaxation phenomenon of high residual compressive stresses in the thin film mainly due to the deposition process and involve the propagation, from an initial buckle, of cracks at the film/substrate interface. They have been extensively investigated in various experimental and theoretical studies [1-5]. However, only a few experimental set-ups have been developed to measure the buckle growth dynamics. In fact, the additional compressive stress can be produced in the adherent thin film by using an experimental apparatus which allows the in-situ observation by AFM of the surface during deformation. Buckling patterns generated during the stress experiments are then described and discussed taking advantage of the high resolution offered by the atomic force microscopy (AFM). It has been observed that buckling structures may evolve from straight-sided wrinkles to either worm-like or varicose patterns when the applied stress is released. The formation of these two structures from the initial straight-sided wrinkles has been characterized. In particular, it has been found that one of the driving forces of delamination is the relaxation of the stresses along the longitudinal axis of the initial wrinkles. In the case of bubbles, the film does not *recombine* with the substrate; in situ picosecond acoustic experiments [2] would allow studying the adhesion between film and substrates in this region of interest. Finite element simulations have been done in order to investigate the delamination evolution [4, 5]. Confrontations with in situ stress mapping measurements have to be achieved for validating and then improving these simulations.

In this work, we proposed to use the Scanning X-Ray microdiffraction technique developed at the ALS to perform in-situ spatially resolved stress measurements [1, 3] during a compression test on a film/substrate set. These measurements are expected to provide new insights on the first stage of the delamination process. Preliminary experiments have been done at ALS on gold thin films deposited on LiF substrates.

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MS28 P02

Synchrotron Laue micro-diffraction: a new beam line project at SOLEIL for phase identification and mechanics of materials Philippe Goudeau[a], Olivier Thomas[b], [a]*LMP, University of Poitiers, France.* [b]*TECSEN, University of Marseille, France*
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Keywords: 2D mapping, grain orientation, in grainstrains

Micro-focused x-ray beams from 1 down to less than 0.1 micron in size have been one of the real success stories of 3rd generation synchrotron x-ray machines (SR), thanks to a large panel of focusing devices for hard and soft x-rays [1-5]. There is clearly a rapidly growing need for very small x-ray beams – 10 nm is theoretically possible - which allow for non-destructive local scale measurements of structure and chemistry. This need encompasses many different scientific fields: Microelectronics and microsystems, Metallurgy and mechanics, Environmental and earth sciences, Art and archaeology, Life sciences and soft condensed matter. In all these different research fields one would ideally like to get information on a local scale of the structure, the chemical composition and the local atomic environment. This implies performing at the submicron scale: XRD, XRF, EXAFS, XANES. However, the case of XRD technique is specific in the sense that the recorded signal does not only depend on the beam size but also on the ratio of the beam size to the grain size. If this ratio is large enough one is left with powder diffraction when using monochromatic x-ray beam (MB) and the diffraction information is an average over the size of the beam. On the other hand if the beam size over grain size ratio is small one gets single crystals diffraction. It is thus possible to obtain intra grain structural information. Transmission configuration with hard x-rays is done using either MB or white beam (WB) XRD with generally energy dispersive mode. For the proposed beam line, the working geometry is reflection. It is important to realize, however, that recording a significant number of diffraction spots from a single crystal requires either a movement of the sample under the beam (goniometry) or the use of a polychromatic incoming beam (Laue diffraction) combined with a two-dimensional area detector such as CCD type. Since there are no available goniometers with a sphere of confusion (SOC) radius smaller than the micrometer x-ray probe size it is thus necessary to use WB. Details concerning the different applications of the technique may be found for instance at the Advanced Light Source (ALS) web site address, this synchrotron source being close enough to the French facility SOLEIL. This beam line project is unique in Europe since there is almost nothing in European countries in terms of fully dedicated WB µXRD beam line except the BM32 project at ESRF (10 % of the full beam line time dedicated to microfocus WB during 3 years). The Scientific Advisory Committee (SAC) of SOLEIL approved the preliminary beam line project APS (document available at CnanoNO web site) in November 2005 and the SOLEIL Council decided in July 2006 to build this additional beam line and to provide ¼ of the total beam line budget which will be completed by external funds (not yet found).

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MS28 P03

Combining microRaman and microdiffraction to investigate keratin modifications during in situ deformation Emilie Leccia*, Richard J. Davies°, Fatma Briki*, Jean Doucet*, **Laboratoire de Physique des Solides, Université Paris-Sud, France.*
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Keywords: microdiffraction, Raman spectroscopy, keratin

The structure of keratin still raises many questions, even though it has been studied for more than 50 years. This is partly because the fibrous protein cannot crystallize, and therefore its structure cannot be determined at atomic resolution. X-ray studies of keratin are typically carried out as fiber diffraction experiments, and usually on keratin in its original tissue form such as hair or epidermis. The advantage of this method is that it gives rise to results spanning both atomic and supramolecular length-scales. However, although X-ray diffraction can provide a range of structural information, it is inherently limited for studying amorphous materials. In contrast, Raman spectroscopy is capable of probing both the amorphous and crystalline fractions of a material, and is therefore somewhat complementary. For example, Raman studies of keratin provide access to structural information concerning the secondary proteins (i.e. random, beta-sheet, alpha-helix).
Several previous studies have attempted to characterize the structure of hair keratin during macroscopic deformation. Nevertheless, questions still remain as to whether stress-induced transitions from alpha-helix to beta-sheet can occur. The complementary information provided by Raman spectroscopy and X-ray diffraction is ideal for answering this question. In addition, both methods are compatible with in situ deformation and are available using microfocussed beams. This means that single hair fibers can be deformed in situ. Not only does this reduce structural averaging compared to the use of fiber bundles, but it also makes the stress calculation more accurate. Moreover, diffraction and Raman data collected simultaneously on a given sample minimises the risk for artefacts due to sample preparation and biological variability.
This study reports on the innovative coupling of simultaneous microdiffraction and microRaman spectroscopy for investigating the deformation of hair fibers. This capability is available at the ID13 beamline of the European Synchrotron Radiation Facility [1]. The system uses a pierced mirror to deliver the laser and X-ray beams coaxially to a common focal position on the specimen. The spot size is approximately 1 µm for both the laser and X-ray beams. The results indicate that the diffraction peak at 5.15 A disappears during macroscopic deformation. This can be correlated in the Raman spectrum with a bandshift assigned to the C-C bond in the alpha helix skeleton. These observations are interpreted in terms of the unraveling of alpha-helices during stretching. Neither the Raman nor X-ray data show any evidence of beta-sheet formation. This suggests that although the deformation of hair causes molecular distortions, there is no alpha-helix to beta-sheet transition (under the current experimental conditions).

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MS28 P04

Morphological and structural characterization of nanomaterials in forensic science Marek Kotrly[a], Veronika Grünwaldova[b] [a]Institute of criminalistics Prague, Czech republic, [b]Zentiva a.s. Czech republic.
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Keywords: forensic microanalysis, nanocomposites, phase analysis

Nanotechnology is among one of the most dynamic and constantly developing fields of current material sciences. The contemporary level of scientific knowledge, procedures and technologies in the sphere of nanotechnologies can be compared to the situation emerged in electronics, PS technologies and in telecommunications in late forties and early fifties of the 20th century, shortly after transistor invention. Similarly, the field of forensic science does not keep aloof, materials containing nanoparticles and nanocompozites are increasingly encountered with performing forensic/technical expertise. The majority of material analyses in forensic field deals with material comparison and phase analysis. Nanoparticle occurence presents valuable information for establishing of identity of objects, for assessing the origin of the product, etc. The complex analysis comprising electron microscopy, namely TEM, FE SEM (field emission SEM) technique is usually used for materials embracing nanoparticles enabling to scrutinize the morphology if surface particles, FIB (Focused Ion Beam) allowing by means of accurate sections to carry out the verification of physical inner structure of multilayer particles and nanocomposites. To obtained image information are applied the image analysis (particularly exact measuring and morphological analysis using the method of mathematical morphology) and last but not least XRD methods. If necessary, AFM technique can be used as well.
Powder diffraction is used both for basic phase analysis and for the identification of nanoparticles, or more precisely for nanolayers. Performing these analyses both issues concerning sample preparation and detection limit are coming to the foreground. Detecton limits regarding nanoparticles content were tested using not only testing materials containing amorphous fillers, but also compounds of profoundly diffractioning phases. The classical arrangement applying Bragg-Brental optics and spotlight scintillation detector coupled with micro-diffraction monocapillary primary optics and linear multi-channel detector (X´ PertPRO) was tested.

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