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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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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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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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