shapes and lattice defects were evaluated from the X-ray pattern. In those cases the determined quantities are characteristic to a large volume of sample.

In the present case the X-ray diffraction technique was employed for characterization of individual nano-belts. The measurements of X-ray diffraction lines from a single nano-belt were achieved by using the unique nano-diffraction technique described in [2]. The results were compared with those obtained from SEM/TEM.

[1] Zhao M. H., Wang Z.L., Mao S. X., *Nano Letters*, 2004, **4**, 587. [2] Xiao Y., Cai Z., Wang Z. L., Lai B., Chu Y. S., *J. Synchr. Rad.*, 2005, **12(2)**, 124.
Keywords: nano-belts, X-ray nano-diffraction, nano-structure

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Structural Characterization of Hybrid Carbon Nanomaterials
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Reflection High Energy Electron Diffraction (RHEED) has been used to investigate the structural features of a new class of nanostructured carbon materials, coupling nanosized diamond with single-walled carbon nanotubes. This innovative material is being produced in our laboratories in a modified CVD reactor by means of reactions between carbon nanopowders and atomic H.

We investigated samples grown at increasing deposition time, combining the structural RHEED data with the information achieved by complementary analysis techniques (Field-Emission Scanning Electron Microscopy (FE-SEM), transmission electron microscopy (TEM), Raman spectroscopy) and by a suitable theoretical approach using *ab initio* modelling [1].

We have been able to determine the growth sequence of the carbon nanophases and the architecture of the observed hybrid nanostructures. Their inner structures are found to be single-walled Carbon nanotubes (SWNT) or bundles of them, and the outermost deposit consist of faceted diamond nanocrystallites.

The experimental conclusions confirm the theoretical prediction [1] about the role of atomic hydrogen in creating localized sp3 hybridized defects on the outer wall of carbon nanotubes, able to promote the formation of suitable sites for nanodiamond nucleation.

[1] Barnard A.S., Terranova M.L., Rossi M., *Chem.Mater.*2005, 17, 527.
Keywords: RHEED, nanophase systems, carbon nanotubes

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Strain, Size and Composition of Buried GaN Quantum Dots in AlN Using Grazing Incidence Anomalous Diffraction
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Structure determination of buried nano-structures represents a challenge due to (i) the nanometric scale of objects and (ii) the presence of strain fields, which produce a 3D (non-discrete) diffuse scattering.

We have developped the use of grazing-incidence multi-wavelength anomalous scattering, which allows to extract the scattering contribution of the (resonant) atoms only. By targetting the resonant edge of one atom of the nano-structures, it allows solving the sub-structures of the nano-objects without requiring any model or prior information.

We will show how this technique can be used to extract the sub-structure of GaN Quantum Dots (QD) in AlN, to obtain the size and strain of QD as a function of the number of layers of QD deposited.
Keywords: nanostructures, anomalous scattering, synchrotron

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Structure Indexing and Solution from Non-Ambient XRPD Data
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Despite excellent recent progress, crystal structure determination from powder data remains a challenge.[1] This is especially so for flexible and weakly diffracting organic (inc. pharmaceutical) compounds.[2] Of particular interest to the pharmaceutical industry is the full structural characterisation of crystalline forms under both ambient and non-ambient conditions. This arises from a desire to understand the behaviour of solids, and how the materials respond to a range of humidities and temperatures.

The necessary diffractometer hardware to conduct non-ambient experiments (*e.g*. an Anton Parr humidity stage or a TTK temperature stage) is well established,[3] but the application of such experiments to pharmaceutical compounds is less so.[4] Here we present the methodology required to obtain high resolution non-ambient data for crystalline forms that correspond to phase changes observed in other solid state analytical techniques (*e.g*. DSC or GVS). We have indexed and subsequently solved the structures of observed forms from this non-ambient data to give full structural information across the phase diagrams. In addition, the thermal expansion coefficient for the material can be determined by indexing over a range of temperatures. This value is key for the construction of a pressure *vs*. temperature thermodynamic phase diagram.

[1] a) see, for example, David W. I. F., Shankland K., McCusker L.B., Baerlocher Ch., *IUCr Monogr. Crystallogr.*, 2002, **13**, 337; b) Harris K.D.M., Cheung E.Y., *Chem. Soc. Rev.*, 2004, 33. [2] Shankland K., Markvardsen A.J., David W.I.F., *Zeit. Krist*., 2004, **219**, 857-865. [3]see, for example, Anton-Parr-Str, Graz, Austria: website www.anton-parr.com . [4] Brittain H.G., *Spectroscopy*, 2001, **16**, 14-16.
Keywords: powder diffraction under non-ambientconditions, pharmaceutical structure determination, hydrates

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Structure Analysis of Pharmaceutical Compounds from Powder Diffraction Data
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In recent years, the crystal form of pharmaceutical compounds has become important not only for identification of the quality but also for accession of the patent. However, powder diffraction data using standard in-house instruments has low resolution as compared to the synchrotron radiation data. Accordingly, we have been studying the usefulness of the synchrotron radiation and have identified the crystal form of pharmaceutical compounds. In this study we investigated polymorphs of carbamazepine, taurine and acetaminophen as an example of pharmaceutical compounds. Additionally, we made a study on the structure determination of carbamazepine and taurine using the synchrotron diffraction data.

All pharmaceutical compounds, except for carbamazepine form III, were purchased from Wako Pure Chemical Industries (Tokyo, Japan). Carbamazepine form III was prepared by heat treatment of form I at 443 K for 2 hrs. The concomitant samples of carbamazepine were prepared by mixing form I in form III with mortar and pestle moderately. Powder X-ray diffraction patterns were collected with BL24XU of SPring-8 using milled powder samples packed in a quartz glass capillary.

As the result of analysis for concomitant polymorphs of carbamazepine, the peaks of 0.5 % form I at 12.06° and 12.3° (2Theta) are detected. By Rietvelt refinement, Rwp of the carbamazepine (form III) is 6.00 % (Rp=4.02 %).
Keywords: pharmaceutical compounds, polymorphs, powder refinement