Commercial Lecture

THE 1DER DETECTOR FOR EMPYREAN X-RAY DIFFRACTION

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In an X-ray diffraction measurement, fluorescence from some elements can be strong enough to create an unwanted background in the measured data. With the unrivalled performance of the new 1Der detector, removing unwanted fluorescence background has just become better, faster and easier. A major advantage of the 1Der over previous detectors is its superior energy resolution. With this new level of performance, it is possible to eliminate more fluorescence interference than ever before. The narrow bandwidth of the 1Der, together with Empyrean's unique incident beam optics, excludes all unwanted fluorescence energies from a measured scan.

Crystallography and especially crystal structure refinement are vital components in the efficient discovery and development of new materials in all sectors. Crystal structure refinement is required in many advanced materials where a clear understanding of the stability of crystal phases is necessary for control over significant materials properties. The drive to improve crystal structure refinement is more significant than ever as the sophistication of advanced materials increases. The Empyrean system with the new 1Der detector provides the highest sensitivity X-ray powder diffraction data available today.

The 1Der is compatible with all the X-ray sources that are used on the Empyrean. With all Empyrean X-ray tubes, users can quickly change the source themselves. This enables them to select an improved performance for unique applications. A multisource system opens the possibility to measure applications previously inaccessible on a single source system.

As an example, soft radiation (e.g. Cr) can be used to increasing the diffraction angle in an experiment and hence improve the resolution of closely space peaks, such as those seen in clays and ceramics. When used in combination with higher energy or 'hard' radiation (Mo or Ag) the 1Der detector can be used for example to to measure total scattering data suitable for Pair Distribution Function (PDF). Hard radiation is also used to penetrate solid objects such as metal components or pouch cell batteries and is used in transmission experiments.

POSTERS

P1

INFLUENCE OF INTERNAL STRUCTURE ON PROPERTIES AND MORPHOLOGY OF NANOFIBER TEXTILES

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Our previous results showed that crystallization in the strong electric field, especially in case of wire spinning affects the crystal structure and phase composition of nanofibers. In any case the crystal structure is strongly affected by the preferred orientation of crystallites. The texture is typical fiber texture with cylindrical symmetry of distribution function of crystallites orientation. Fiber axis is in polymer chain axis and all the crystallographic planes, which are not parallel with texture axis, are suppressed in XRD powder diagram. This makes the structure and phase analysis very difficult. Anyway the profile analysis of diffraction pattern showed clearly that electrospinning changed the phase composition especially in case of wire spinning.

In present work we have found two special cases: PAN and PVDF, where the specific crystal phase in electrospun nanofibers significantly affected the fiber morphology and properties. In both cases electrospinning led to dominant crystal phase, that is characterized by two common features: (1) layered arrangement of chains in crystal structure and (2) charge polarity and electroactivity of these layers, as one can see in the figure 1. Both of these effects lead to the flat shape of crystalline fibers in the form of thin strips, which due to their flexibility and charge distribution roll up into tubes - forming hollow fibers. This work showed that hollow nanofibers can be prepared in a simple way and thus produce a nanofibrous membrane with a higher surface for further chemical modifications, respectively to improve the sound-insulating properties of the membranes.

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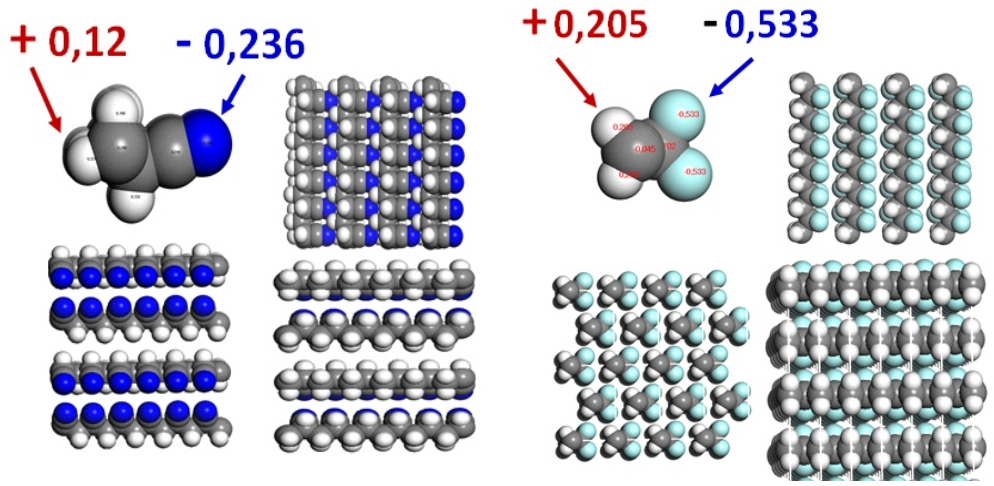
(figure on next page)



a) b)

Figure 1. Illustration of charge distribution in polymer chains for PAN (a) and PVDF (b) in upper left corner (a) and (b). Charges were calculated in Materials Studio modeling environment. In the upper right corner of the images is the top view of one layer of polymer chains. Below are two perpendicular side views of the layered structures PAN (a)

P2

RUTINOSIDASE – LABYRINTHINE JOURNEY FOR X-RAY STRUCTURE

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Obtaining well diffracting crystals and solving protein structure can be tedious work and successful process may include various crystallization techniques and tricks. Here we present one didactic story of crystallization -**L**-Rhamnosyl- -**D**-glucosidase (Rutinosidase) from *Aspergilus niger*. During the crystallization process, we performed screening using vapour diffusion method, optimization by counter diffusion technique, and final crystals soaking of heavy atoms in micro batch experiments, which allowed structure solution by SIRAS. However, to repeat the crystal growth, we had to deglycosylate the enzyme and perform new screening followed by Matrix Microseed Screening. Moreover, as final reproducible procedure, for growing the protein crystals, we used under oil micro batch experiments. With this optimised method, we were able to grow crystal that diffracted up to 1.27 Å resolution and see structural details that shall be used in the future.