Conceptual Design Report for DANMAX

In situ materials studies in the 10-35 keV range using powder X-ray diffraction and full field imaging.

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1. Summary

DANMAX will be a world-leading materials science beamline dedicated to *in situ* and *operando* experiments on real materials. The beamline will operate in the 10-35 keV range and have two end stations: one for full field imaging and one for powder X-ray diffraction. With a large and diverse user community there will be a focus on high throughput and extended provision of data analysis tools. The combination of two related techniques will ensure cross talk between communities and seed new collaborations and science taking advantage of the high complementarity of the techniques.

The *imaging end station* will enable multi-modal, multi-scale analysis of the internal structures of bulk materials and objects. It will combine absorption/phase contrast, diffraction contrast, and grating-based imaging on medium sized (0.1–5 mm) samples. It will be explicitly designed to enable acquisition of time-resolved 3D movies of structural evolution with a 3D spatial resolution in the range 50 nm–5 μm. As such it will allow direct observation and quantification of material responses to external loads, e.g. during mechanical, thermal, electrical or chemical loading.

The imaging end station will exploit the unique source characteristics in three ways:

- The higher coherent fraction of the light will lead to better phase contrast images.
- At a fixed exposure time, the superior brilliance will enable movies to be acquired with a higher spatial resolution (and higher contrast) than at existing beamlines.
- The increased number of photons on the sample will allow comprehensive diffraction contrast mapping to be performed in minutes rather than hours, vastly expanding the options for *in situ* studies and making combined phase/diffraction contrast studies easier.

The *powder X-ray diffraction end station* will use the latest generation of curved pixel strip detector and the latest generation of large 2D pixel detectors for high-speed *in situ* experiments. The curved pixel strip detector will have a large angular coverage, which will facilitate e.g. pair distribution function (PDF) measurements. The 2D pixel detector will be movable and can be positioned at different distances from the sample. Placed close the sample the 2D detector allows high Q range coverage at moderate reciprocal space resolution. Alternatively the 2D detector can be moved further away from the sample to increase peak resolution at the expense of Q range coverage, or to enhance the low Q region. Both configurations are compatible with very fast data collection, texture analysis, and diffraction tomographic data that benefits from the very small and high brilliance beam at MAX IV.

The station will exploit the unique source characteristics in the following ways:

- The small emittance of the beam will lead to high angular resolution, thus minimizing instrumental broadening. This will allow experiments on more complex samples and to rapidly obtain high quality data of unsurpassed quality.
- The small divergence and the small pixel size of the detector imply that there is no need for analyzer crystals to do high resolution diffraction. The energy discrimination of the detector can be set to suppress fluorescence background.

- The high intensity combined with the fast readout of the large 2D pixel detector will enable a superior time resolution for *in situ* experiments. The rapid data collection and the small beam also allow for point beam diffraction tomography data collection.
- The high intensity will allow smaller sample volumes to be used. Hereby minimizing systematic effects, e.g. absorption and extinction.

The *DANMAX user consortium* (listed as an Appendix) comprises 48 university staff members, representing nearly one half of the synchrotron radiation user's community in Denmark. 18 major Danish companies are also represented. Full service will be possible via the involvement of two industry portals, dedicated to making imaging and powder X-ray diffraction more approachable for the private sector. Building on substantial past experience, instruments and advanced sample environments will be designed and built in Denmark, while analysis tools will be provided through the incorporation of large Danish scientific computing and visualization groups. The Danish groups will staff the beamline.

The *technical design* is at the time of writing incomplete. In particular the technical and economic impact of simultaneous use of the two stations needs further study. A main design choice is to avoid the expense of a satellite building and invest in the undulator, sample environments, and detectors instead.

2. Project Background

While Sweden has a strong tradition in spectroscopy, the Danish X-ray community has its main strengths in diffraction and imaging. The unique properties of the source and the proximity of the site have from the onset attracted strong interest in MAX IV within these Danish communities. The concept for the DANMAX beamline is a beamline that combines two powerful techniques to create a unique instrumental platform to do experiments on *real materials*, under *real conditions* in *real time* at MAX IV. In other words; the project revolves around chemistry, materials science and engineering.

This led to the submission of the DANMAX proposal for the National Danish Infrastructure Roadmap in 2010. During prioritization, the proposal was postponed with the argument that an upper level Danish-Swedish understanding was not in place. Later discussions between the Danish and Swedish ministries had the outcome that Danish participation in the funding of MAX IV indeed would have to take place at the university level and most likely in terms of co-funding of a beamline.

During 2014, the DANMAX consortium has secured grants of approximately 100 million Swedish kroner from the "Nationale Udvalg for ForskningsInfrastruktur (NUFI)", from the Danish regions, and from the universities themselves. The beamline proposal was presented at the "MAX IV User Meeting" in September 2014, and presented to the MAX IV board on October 2, 2014. The board gave a favourable feedback, subject to the evaluation by the SAC.

2.1. Relation to Swedish Users

With respect to *imaging,* the "MAX IV Imaging Workshop" in 14-15 May 2013 revealed a strong overlap between the needs of the Danish and Swedish user communities. The joint recommendation was to develop two complementary beamlines for non-medical imaging in the 10-35 keV regime:

- NANO-MAX. Imaging at the ultimate spatial resolution will be on small samples, relying on scanning an X-ray nanobeam and Coherent X-ray Imaging (CXI)/ptychography approaches.[1]
- For studies of bulk materials, the preferred solution (by all attendees) was to use full field multi-modal and multiscale imaging approaches.

The two communities have therefore coordinated their efforts.† The instrument specifications in the i-MAX CDR are identical to the one proposed here as part of DANMAX, and the science case presented by the Swedish users is aligned with the science case here.

Regarding *powder X-ray diffraction* the two proposed beamlines DANMAX and DiffMAX looks at first glance similar, however DiffMAX is intended for GISAXS, powder, single crystal, and surface diffraction significantly reducing the available time for powder diffraction. While the fundamental principles of powder X-ray diffraction beamlines are similar, differences in sample environment and beam parameters make each one especially suited for different experiments. DANMAX will focus on the core competence, which is *in situ* raction under *real* conditions.

3. Scientific Case and User Community: Imaging

In this and the following chapter we present the science case and examples of use for the two communities. It should be noted that there is an overlap, and imaging activities may be presented under powder X-ray diffraction and vice versa.

X-ray tomography allows 3D mapping of internal structures of materials without destructive sample treatment. This is invaluable, for example, when studying paleontological, archaeological and cultural artefacts that are precious and often unique, such that destructive analysis is not an option. A more general area of application is 3D characterization of material microstructures over representative volumes. Examples are the characterization of pore structure and connectivity for fluid flow modelling in porous media or understanding the morphology and juxtaposition of material microstructures that control macroscopic deformation. Furthermore, the ability to observe materials under the correct environmental conditions, and as a function of time, is often essential.

DANMAX will provide a number of unique or improved options for imaging:

- *Improved contrast and resolution*. The aim is not to set new records with respect to time or spatial resolution. However, the increased number of photons on the sample, and the increased coherent fraction, implies that for a *given acquisition time* the spatial resolution and in particular the contrast of phase contrast images will be superior.

† The editors acknowledge numerous discussions with Steve Hall, LU, and parts of the text here are copied from the iMAX proposal. Also technical discussions with Ulrich Lienert, responsible for the Swedish beamline at DESY, are much appreciated.

- *4D materials science.* This field has emerged within the last decade.[2] Here the synchrotron is used to generate 3D movies of materials behaviour. The initial map is used as input for 3D modelling. In this way two movies – an experimental and a simulated one – can be compared point by point and time-step by time-step. This is seen as a much-improved route towards establishing and validating materials models. The increased number of photons on the sample and the improved contrast will facilitate 3D movies acquired at a much reduced acquisition time.

- *Multiscale imaging.* It is characteristic that materials are organized hierarchically. Hence, the option to "zoom in and out" in the material is a major asset. DANMAX will first of all provide absorption and phase contrast imaging with high spatial resolution, permitting fine scale studies of bulk material microstructures. Combinations of these techniques with local tomography or grid-based dark-field imaging[3] will enable multiscale analyses of structures and processes.

- *Multimodal imaging.* Another key focus of DANMAX will be multimodal imaging and, in particular, exploitation of full-field diffraction contrast imaging. A large group of materials are polycrystalline, e.g. most metals, ceramics, rocks, ice, sand and soil. Using diffraction based methods such as 3D X-Ray Diffraction (3DXRD)[4] and Diffraction Contrast Tomography (DCT)[5] the evolution of the individual grains and domains can be followed, as well as their orientation and stress state. On the sub-micron scale point beam methods allow distinguishing and mapping nanocrystalline and amorphous constituents[6] as well as following their response to e.g. external load. In a similar way to phase-contrast imaging this can also provide enhanced contrasts in low attenuation matter. In all cases it is a major science driver that the improved brilliance allows the acquisition time for diffraction based maps to be much lower, such that the gathering of multimodal data can become routine operation.

Below we provide several examples of the Danish science drivers for the imaging end station

Composite Materials for Wind Turbine Blades
B. Sørensen, DTU Wind; Industry: LM, Vestas, Siemens Windpower.

The load carrying part of wind turbine blades are based on strong uni-directional fiber reinforced composite materials where the key material properties are high stiffness, high compression strength, and good fatigue damage resistance. Mastering those properties, it is possible to make longer and/or lighter wind turbine blades with the effect of lowering the cost of energy from wind turbines. Based on X-ray tomography, it is possible to visualize the fiber architecture and fatigue damage evolution. Details in the fiber architecture have a key effect on the compression performance[7] as well as the fatigue damage evolution.[8] In addition, by studying the fatigue damage evolution in itself it is possible to understand the mechanism behind tensile fatigue damage in the relevant type of composites. Such studies require a high brilliance source, in particular because phase contrast typically is required.

Successful synchrotron experiments have already been done in,[9] where the local damage development during static testing was monitored. Easier access and a brighter source will make it possible to harvest the full potential, including *in situ* studies of damage.

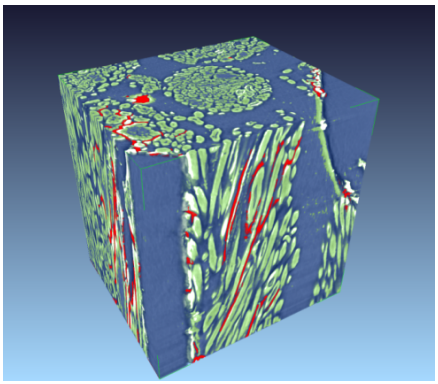

Figure 1: 3D representation of fibres in a composite material for use in wind turbines.

Energy Conversion and Storage
L. Theil-Kuhn, J. W. Andreasen & J. R. Bowen, DTU Energy.

For applications such as catalysts, fuel cells, and ammonia storage, it is vital to understand the flow processes of gases in porous media. Today, performance is mainly investigated on the macro scale, and a microscopic understanding of the flow/performance relationship is lacking. As an example, there are simple ways to determine the overall "porosity" but the gas/fluid permeability cannot be predicted with any accuracy without knowing the spatial distribution of the pores and their topological connection in 3D. Uniquely, X-ray imaging enables input of the real complex microstructure into computational fluid dynamics (CFD) models. In some cases, we combine this with phase field modelling to predict the porous microstructure evolution, e.g. during degradation. Furthermore, by using fluids with embedded marker nano-particles, it will – under favourable conditions – be possible to observe the flow directly in 3D.

Fuel cells: A major factor controlling the performance and durability of solid oxide fuel cells is the electrode microstructure, i.e. transport of gas in porosity and electrons/ions in solid phases to and from reaction sites. Changes in solid phase morphology during fabrication and operation directly influence reaction site density, pore connectivity, gas flow and thus performance. In the CINEMA alliance we follow electrode *in situ* evolution as a function of temperature, atmosphere and ultimately *operando* as a functioning fuel cell under current. We use our expertise from electron microscopy[10] to quantify 3D movies, which will be compared to 3D phase field simulations of electrode microstructure evolution. The proposed beamline capabilities (phase contrast at high energies and high resolution ~50 nm) are required for breakthrough characterisation of high temperature energy devices where high penetration through transition metal compositions is necessary such as recently demonstrated at the ESRF.[11] Unique *in situ/operando* experience and sample environments developed in CINEMA will thus be ready for exploitation with the availability of DANMAX.

Energy storage: Typically loading and un-loading of energy storage media is associated with major microstructural changes. Regions of the media may either not be saturated at all or take too long to do so, i.e. reducing efficiency or prolonging refilling times. Uniquely, 3D imaging can monitor such processes during operation. As an example, we consider solid-based ammonia storage systems for NO_x reduction in diesel engines. During operation, the material goes from being highly porous (>70%) to being almost solid (<10% porosity). By coupling 3D imaging techniques with simulation tools the influence of salt properties, refilling conditions and cartridge properties on the nano- to microscopic scale, flow properties and deterioration mechanisms are studied.

3D Domain Evolution and Multiscale Studies of Polycrystalline Materials
H. F. Poulsen & S. Schmidt, DTU Physics; J. R. Bowen, DTU Energy; D. J. Jensen, DTU Wind; W. Pantleon, DTU Mechanics.

The group has developed several methods for 3D mapping of grains, orientations, phases and local stress. Most recently hard X-ray microscopy,[12] which enables 3D mapping of individual crystalline elements on several lengths, e.g. domains within grains, see Figure 1, has opened the door to a powerful approach to establish and test multiscale materials models.

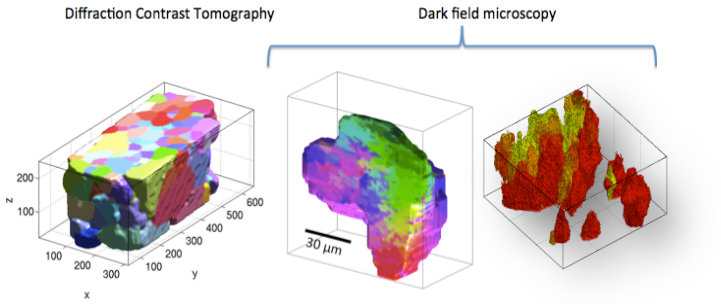

Figure 2: 3D multiscale mapping of deformed Al. Having mapped 1000 grains with the microscope (left) one can zoom in 3D and obtain magnified images of one grain (middle) and on an even finer scale on embedded domains (right).

Multiscale studies will be combined with 3D simulations for understanding and quantitative modelling of grain and domain evolution in real materials. Key science questions are

- Plastic deformation in metals
- Modelling of coarsening
- Interaction between strain and domain evolution in ferroelectrics
- Dislocation dynamics

This approach to materials science is currently being developed in collaboration with ID06 at ESRF. It involves a larger program at DTU for optics development and 3D algorithm development. When DANMAX becomes operational it is anticipated that the microscopy approach is ripe for routine use. At MAX IV, the acquisition times for coarse scale grain maps and fine scale domains maps will be reduced from hours to minutes and from minutes to sub seconds, respectively.

Oil Extraction and Natural Materials Science
S. Stipp & H.O. Sørensen, KU Chemistry; B Vinter & R. Feidenhans'l, KU NBI; Industry: Mærsk, BP, Reykjavik Energy, COWI & Rockwool.

Nanotechniques provide fundamental information about the processes that control the physical and chemical properties at the interface between natural solids and fluids (anything that flows, i.e. water, oil and gases). This new understanding contributes to solving some of society's challenges such as ensuring clean water, safe storage of waste, converting CO_2 to rock, squeezing more oil from spent reservoirs and understanding the mysteries of biomineralisation, i.e. how organisms make bones, teeth and shells. 3D imaging lets us characterise the micro and nanostructure of soil, sediments, rocks and biominerals and observe how they change as a function of stress or exposure to fluids. Applications of imaging include derivation of the petrophysical parameters of chalk, such as porosity, permeability and tortuosity[13] and characterisation of the mechanical properties of a heart urchin[14] as well as direct imaging of multiphase flow (e.g. oil and water) in reservoir rocks.

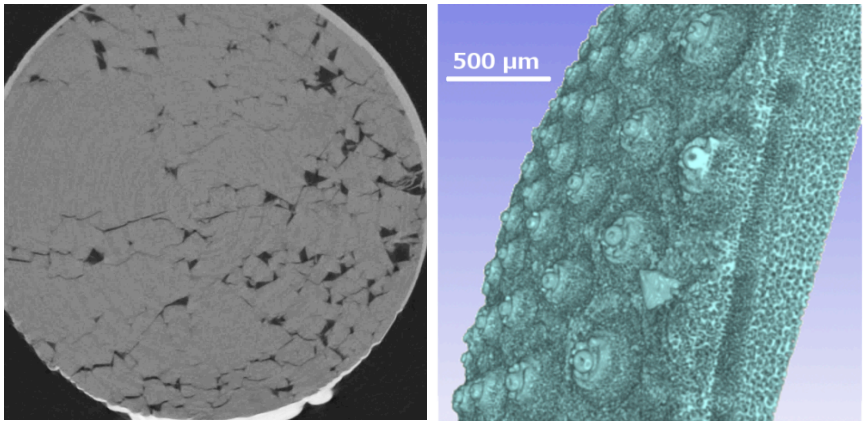

Figure 3: Single slice from a tomogram of chalk (left) and heart urchin shell (right).

Applications of simultaneous diffraction and imaging include identification of minerals present in fractures, which reveal the conditions of their genesis[15] and determination of crystal orientation in biomineralised materials as a pathway for mechanical optimization. Until now, the group has worked at beamlines in Switzerland, Japan and France. The DANMAX beamline will provide access closer to the labs, making sample handling less risky. In addition the superior phase and diffraction contrast will be crucial for many studies.

Applications in Food Science
R. Feidenhans'l, KU NBI.

Product quality and product development is a vital part of the Danish food industries today. Development of new products requires efficient and powerful techniques that assess the components of a product using non-destructive modalities with high fidelity. Three dimensional X-ray imaging with much higher contrast can be provided by phase contrast imaging. This gives a direct measure of the electron density of a material and is very well suited for imaging of tissue and light materials.[16] An example is shown in Figure 4 which shows tomographic slides of porcine fat based on absorption and phase contrast imaging.[17] The results show an encouraging potential of detection of fatty acid composition, high contrast between cartilage and soft bones, even connective tissue may be discernible in meat products.

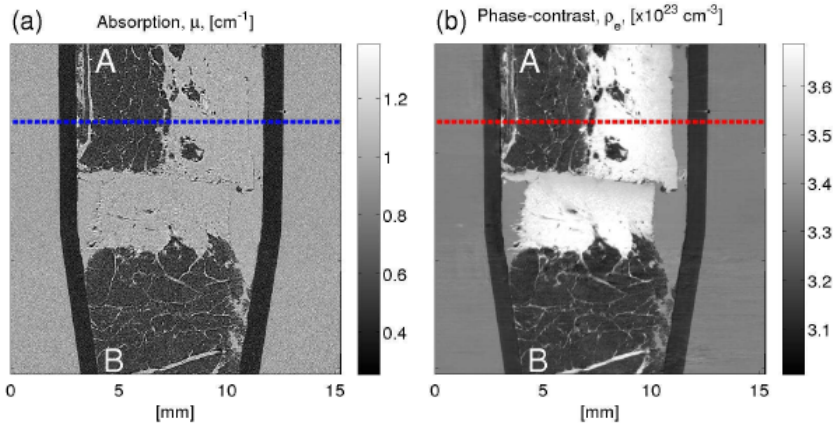

Figure 4: Tomographic slices of porcine fat. (a) Frontal slice through the tomographic reconstruction of the absorption contrast. (b) Frontal slice through the tomographic reconstruction of the phase. The phase gives clearly higher contrast.[16a]

DANMAX will give new opportunities to characterize various quality traits of food products in meat, emulsions, dairy, sweets and fruit and also in development of new advanced packaging materials. For meat, these aspects include tenderness and intramuscular fat distribution, subcutaneous fatty acid composition of porcine fat, and drip loss of porcine meat. With X-ray phase contrast the electron density is directly measured, which might directly be linked to the carbohydrate, fatty acid, and protein composition (proteomics) of the tissues. With DANMAX new possibilities emerge which could allow to follow processes like cooking *in situ* and follow detailed structural changes in real time.

Biological and Bioinspired Materials
H. Birkedal, AU Chemistry.

Biology makes multiphase hierarchical nanomaterials to address a wealth of important materials problems including underwater adhesion, actuation, self-healing materials, high toughness low weight materials etc.[18] Bone is a prime example of this: It is formed from nanocrystalline calcium phosphate and proteins in an incredibly complex composite material.[18-19] Bone structure and its relation to bone performance in both healthy and diseased bone remain far from understood. Upon incorporation of biomaterials into the body, a wide spectrum of poorly understood responses result. The phase tomographic capabilities of DANMAX are particularly useful for the study of biological materials and biomaterials since they often contain both inorganic absorbing and organic weakly absorbing components – both of which are essential for understanding function. The high spatial resolution together with the zoom-in capabilities of DANMAX will allow covering the many length scales needed to understand these materials and lead to improved fundamental understanding and applied biomaterials.

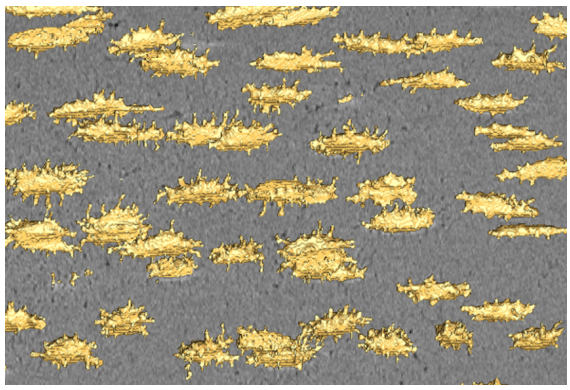

Figure 5: Volume rendering of osteocyte lacunae in bone (yellow) on a background of a slice through a reconstructed tomographic section through a piece of bone. Osteocytes are long living bone cells involved in sensing mechanical stress on bone and in maintaining healthy bone.

The relation between structure and function is in general poorly understood for biological materials and constitutes a very active area of research. A central problem is that tools are missing that allows mapping performance onto structural features. DANMAX with its focus on permitting studies of samples in complex environments will be uniquely well-suited in this regard.

A central problem in understanding the complex biological and bioinspired materials is the sparsity of tools that allow distinguishing the constituent phases and their part in function of the material. Diffraction tomography using small X-ray beams is well suited for such powder-based materials (i.e. where the grain size is small compared to the beam size).[6, 20] At DANMAX such measurements will be well implemented due to the unique combined competences in imaging and

diffraction. The technique allows studying, for example, the distribution of mechanical deformation on the constituent phases. Preliminary experiments on bone indicate that the distribution of strain induced by external load is surprisingly inhomogeneous.[21]

4. Scientific Case and User Community: Powder X-ray Diffraction

Powder X-ray diffraction (PXRD) is one of the most powerful ways to study the atomic structure of microcrystalline materials. The immense intensity of synchrotron radiation combined with the low divergence of the beams allows for more complex materials to be studied while using minimal sample volumes, which minimize systematic error effects such as extinction and absorption. The extremely low divergence of the MAX IV source will further enhance the peak resolution by reducing the instrumental broadening.

The goal of DANMAX is to do experiments on *real* material studied under *realistic* conditions at *realistic* time scales. This means that fast and efficient detectors are needed in addition to advanced sample environments e.g. electrochemical cells, furnaces, cryostats, gas flow and high pressure etc. Another requirement is a large angular detector coverage to capture the full diffraction pattern at once. A high angular detector coverage will also facilitate PDF reconstruction, which is particular advantageous in case of amorphous phases forming during e.g. the formation of nanoparticles.

The available energy range for the beamline is wide and facilitates both fluorescent elemental mapping in the low energy range and collection of accurate diffraction data on heavy elements or PDF using high energy X-rays. Element specific fluorescence combined with position resolved diffraction is a powerful technique to study chemically/structurally graded materials. This technique does require an energy dispersive detector.

As already mentioned in chapter 3, the possibility to do diffraction tomography is another very interesting add-on. This technique is highly similar to imaging and yields a complete 3D picture of the distribution of nano/micro-crystal phases in the sample and can e.g. also be used to map out stress/strain as a function of position in the sample.

Below we provide several examples of the Danish science drivers for the PXRD end station.

Nanocrystal Formation and Growth
B. B. Iversen, AU Chemistry.

A very important new development in powder X-ray scattering is the introduction of *in situ* total scattering studies of solvothermal reactions. In high pressure and high temperature environments the formation and growth of nanocrystals can be followed in real time, and during the past couple of years the group has pioneered this field.[22] The ultimate goal of this research is to derive the mechanisms of nucleation, crystallization, and growth of advanced nanomaterials in order to control their correlated functional properties. Solvothermal synthesis is a flexible, fast, environmental-friendly, and cost-effective technique to chemically produce nanoparticles in solution from laboratory to industrial scale. In general, the performance of functional nanomaterials, e.g. electronic or catalytic properties, is highly dependent on their structure and morphological characteristics such as size, shape, crystalline state and phase. Being able to synthesize materials with tailor-made properties is thus crucial in the development of new applications and technologies. Solvothermal synthesis has a large number of parameters that can be varied to alter the product, but

the mechanisms controlling the particle characteristics are not understood. By means of *in situ* PXRD and X-ray total scattering (PDF analysis) it is possible to probe the short-range, local order as well as the long-range, global order of a material. Hence, the structural transformations during synthesis can be followed all the way from the evolution of ionic complexes in the solvent over amorphous clusters to the nanocrystalline phase.

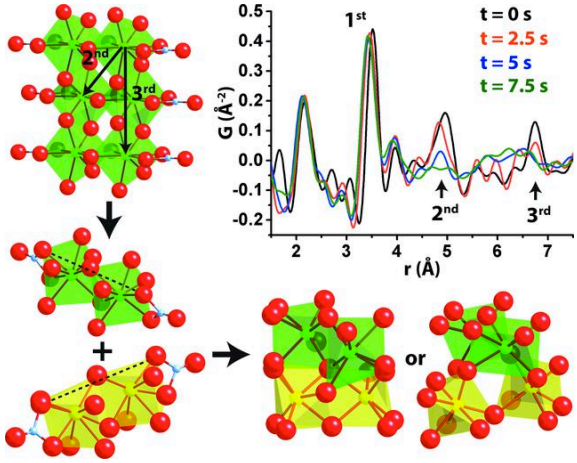

Figure 6: Initial nucleation mechanism for ZrO_2 nanoparticle crystallization under solvothermal conditions.[22d]

The group has developed expertise and instrumentation for *in situ* solvothermal synthesis of nanoparticles and contributed to the modelling of molecular clusters in solution and structural changes on atomic scale during the transition into the crystalline phase under sub- and supercritical conditions. With DANMAX studies of a wide range of new materials become within reach. An exciting new development is to measure PDF data on thin films. Proof-of-principle data has been measured. It is envisioned that within the next couple of years and with the construction of DANMAX this will become generally possible.

Micro Battery Cells for Detailed Structural *in situ* Studies
P. Norby, DTU Energy.

Progress on improved lithium-ion batteries and especially next generation batteries depends on the development of new materials and on an improved understanding of the processes in materials during charge/discharge, the degradation mechanisms and the interface dynamics and reactions. *In situ* PXRD is a powerful method for studying structural and microstructural changes in electrode materials.[23] Development of a capillary-based micro battery *in situ* cell[24] has allowed detailed structural analysis by allowing diffraction information to be obtained from a single active phase in the battery during operation.

The micro battery cell has been used at various synchrotron sources e.g. for studies of structural changes and defect development during lithium intercalation in graphite,[24] phase development in cathode materials for sodium batteries, investigation of reactions in lithium-air batteries, anion intercalation in graphite, interface formation in lithium-ion batteries, and investigations of chemical gradients in electrode layers by micro diffraction.[25] The micro battery cell also allows combined *in situ* experiments with e.g. Raman spectroscopy, XAS/EXAFS and imaging.

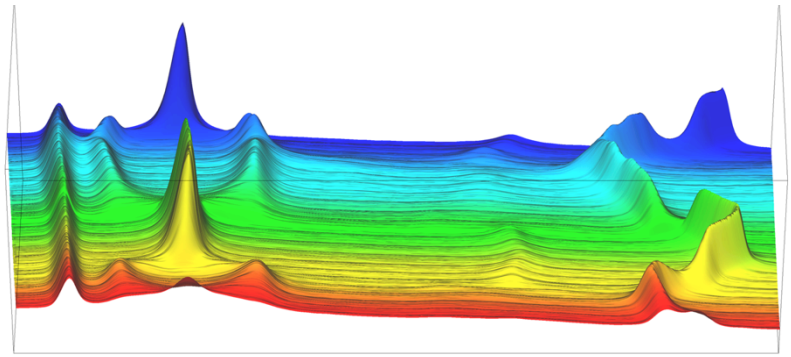

Figure 7: *In situ* powder X-ray diffraction patterns revealing changes in structure and stacking defects during intercalation and de-intercalation of lithium in graphite.[24]

The DANMAX beamline is very well suited for experiments on the micro battery cell, where the high flux and moderate energies allows high quality structural studies with very good time resolution. In addition it opens up the possibility of combined *in situ* imaging and diffraction studies of battery electrodes under operation.

Operando Total Scattering Studies of Battery Materials

D. B. Ravnsbæk, SDU Physics, Chemistry and Pharmacy.

Every time a battery is charged and discharged the electrode materials undergo a phase transition, e.g. in Li-ion batteries a transformation occur between the Li-rich and Li-poor phases. Understanding these transformations and the structural changes they entail is of vital importance as the nature and reversibility of these determines the stability, efficiency, and lifetime of the battery.[26] Batteries inherently operate under dynamic conditions far from equilibrium. Hence, the true nature of the structural changes can only be fully determined when studied during battery operation and at reaction rates relevant for real-life applications.[27] Furthermore, special designed battery test cells are needed, which can deliver high-quality diffraction data as well as high-fidelity in terms of the electrochemistry.[28] *Operando* X-ray diffraction is a highly suitable tool for investigating the phase transitions providing information on the composition, reactions, particle sizes, strain, and indirectly also the mechanism – that being for both crystalline-crystalline (PXRD) or crystalline-amorphous (PDF) transitions.[29]

However, the requirements for the high rates and the special designed sample cells makes the utilization of synchrotron X-rays a prerequisite. The X-ray scattering capabilities of DANMAX will be perfectly suited for *operando* diffraction studies of structure and phase transformations in batteries of both crystalline and amorphous materials during fast charge and discharge cycles.

Moreover, the imaging capabilities at DANMAX will open up for probing the microstructure of electrode materials during operation, which will provide novel insight into charge-distribution, phase segregation, and even phase transformations in single particles. The combination of diffraction and imaging at DANMAX is foreseen to become an unprecedented powerful tool for *operando* battery studies.

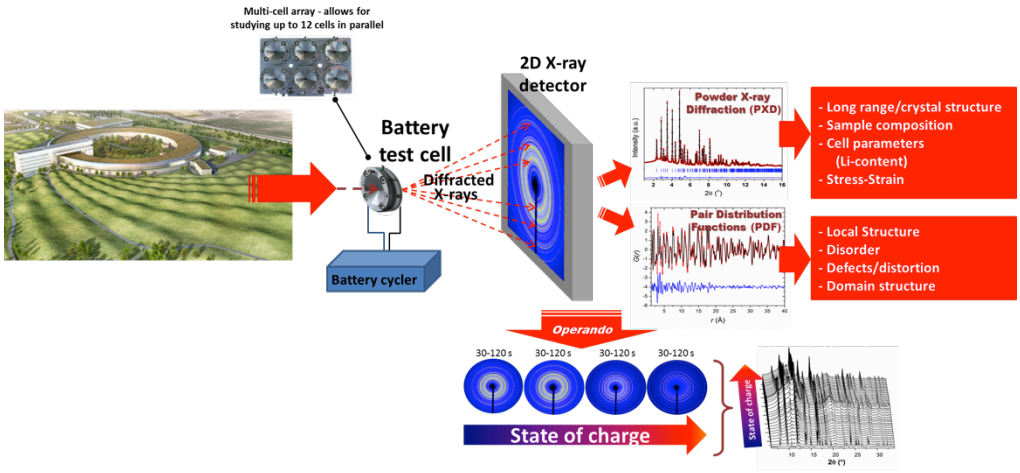

Figure 8: Data collection and data reduction strategy for *operando* total scattering battery studies.

Hydrogen Storage Materials: Identification of New Compounds and Their Reactivity

T. R. Jensen, AU Chemistry.

Synthesis of new materials is an essential aspect of materials science and these are often powders or composites. Novel materials with new functional properties often form the backbone in emerging energy technologies.[30] *In situ* synchrotron studies at variable temperature and pressures are essential for characterization of structural, chemical and physical properties of crystalline solids as well as polymorphic transformations. Chemical reactions between crystalline matter and other solids, liquids or gasses may also be explored regarding the detailed mechanism and the kinetics of reaction.

Systematic synthesis combined with characterization using variable temperature *in situ* synchrotron radiation PXRD (VT SR-PXRD) has successfully been used as an experimental screening approach in order to discover novel materials. Simultaneously, unique information about material composition, structure and properties such as chemical reactions and thermal decomposition pathways has been obtained. The resulting materials were characterized by time-resolved VT SR-PXRD studies, i.e. a three-parameter space is mapped: composition, reactant ratio and temperature.

Products obtained by mechanochemical synthesis are often mixtures of several compounds, which tend to hamper their identification. The SR-PXRD data analysis has demonstrated that new unknown compounds can be structurally characterized also when they only exist as a minor fraction of the samples. The use of multiple powder diffractograms allows for solving structures of two or more new compounds present in the same sample by a method denoted “decomposition-aided indexing and structure solution”. The method depends on good quality diffraction data measured at variable temperature, which allows grouping diffraction peaks according to their behaviour e.g. by peak intensity change or peak shift owing to decomposition, melting or a chemical reaction in the sample. Each group of reflections can be associated with a known compound or used for indexing and structure solution.[31] The energy range, high flux and low instrumental broadening at DANMAX will facilitate more accurate experiments with better time (i.e. temperature) resolution.

Crystal Induced Selectivity: Oxygen Storage Materials, and Other Solid-State Reactions
C. J. McKenzie, SDU Physics, Chemistry and Pharmacy.

Crystalline metal-organic materials can reversibly and selectively chemisorb O_2 from the air.[32] With a range of affinities within the useful Hemoglobin-Myoglobin span, these materials might find application for new types of life-support systems, and other devices requiring O_2 separation and upconcentration from gas mixtures and air. Single crystal structures have been obtainable only in a few cases, namely for the thermodynamically stable oxy forms. In general, however, the materials are weakly diffracting, impeding analyses using lab equipment. The crystallinity of one of the unstable deoxy forms was ascertained by synchrotron PXRD (Figure 9 left). On exposure to air O_2 is re-chemisorbed at varying rates, depending on the electronic properties of the organic components, and the crystal phase. The *in situ* PXRD capabilities of DANMAX will allow the study of structural changes under various gaseous mixtures, at various temperature and pressures. Questions like: 'Is there cooperativity between the active O_2 binding sites?' will be addressed. Mechanistic studies on these systems, and on reactive, catalytically competent, crystalline solid-state metal-oxidant adducts[33] will be pursued. Similarly obtaining structural information on polymorph- and temperature- and guest-dependent magnetic properties of organometallic iron compounds[34] (Figure 9 right) will be enabled.

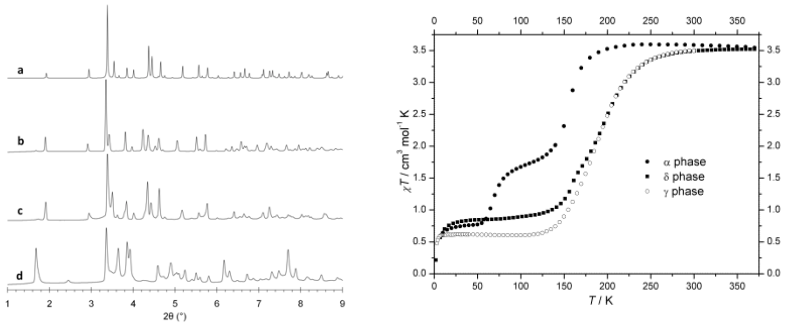

Figure 9: (Right) PXRD patterns measured on 12-BM at the Advanced Photon Source at Argonne National Laboratory (20.05 keV, 0.6183 Å) (a) oxygenated material, simulated from the SCXRD structure, (b) oxygenated material (hydrated form), (c) oxygenated material, and (d) deoxygenated material.[32a] (Left) Magnetic susceptibility of three polymorphs of a magnetically bistable crystalline material.[32a]

Functional Magnetic Materials Perturbed by Guest-Host Interactions
J. Bendix, KU Chemistry.

Functional materials experience an increasing interest in chemistry, physics and materials science. The use of bottom-up approaches has allowed researchers to design coordination-chemistry-based systems in a hierarchical fashion with specific physical properties imposed by the employed molecular building units. Of particular interest are rigid, porous networks incorporating metal ions as "joints" and organic molecules as "linkers".[35] Such materials, commonly referred to as porous metal-organic frameworks (MOFs), are promising candidates for e.g. gas capture/storage and separation[36] as well as heterogeneous catalysis.[37] The performance of a porous material is typically judged by its pressure-dependent gas-uptake but studies of structural evolutions upon application of pressure are nearly non-existent. *In situ* PXRD is a perfectly suited technique for such studies, in particular in our systems featuring exposed fluoride sites that interact strongly though hydrogen bonds[38] and this technique provides the only real possibility to study any changes to the crystallographic lattice as a function of guest molecule pressure. The high intensity at

DANMAX combined with high peak resolution will allow detailed studies of these effects. Furthermore, such investigations form the basis of understanding magnetic perturbations in the novel class of slowly relaxing paramagnetic MOF materials by correlation of structural and magnetic properties coupled to the guest-host interaction.

Textured Magnets from Tailored Magnetic Nanoparticles
M. Christensen, AU Chemistry.

The R&D on new stronger permanent magnets is focusing on nanocomposite magnets of mixed hard and soft magnetic materials. *In situ* compaction give access to information about crystal growth, phase transformation and texture effects during compaction, which are essential for understanding new permanent magnets. By controlling the shape of nanoparticles it is possible to order the crystallite orientation during compaction. This is of special interest in magnetic systems, where the magnetic easy axis can be aligned using a direction mechanical force field. Studying texture effects are of interest in a many crystal systems, where the physical properties are anisotropic along the different crystallographic axis. Other examples are piezo electrics, non-linear optical crystals, superconductors, thermoelectrics etc. In addition to applied mechanical force, nanoparticles can also be aligned through magnetic or electrical fields. A large 2D area detector is essential for capturing the texture information and small beams as provided by MAX IV will facilitate the use high magnetic and electric fields as well as high pressures in diamond anvil cells. The DANMAX beam line would allow following both phase evolution, nanoparticle size and texture simultaneously.

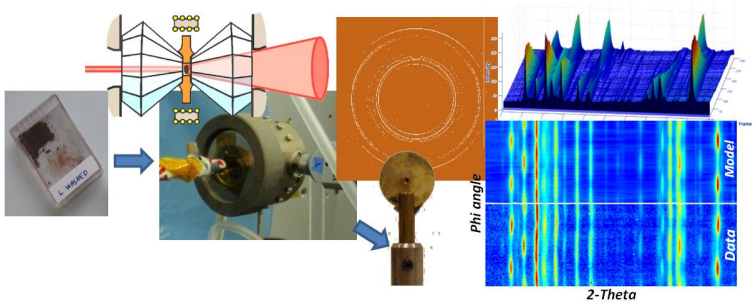

Figure 10: Alignment of magnetic nanoparticles using a diamond anvil cell. The sample texture is measured after the experiment *ex situ.*

Investigating compacted samples by imaging techniques would be highly interesting as direct observation of grain orientation could be obtained. Combining diffraction and imaging would be especially interesting in composites systems, where different magnetic materials are mixed to tailor the magnetic properties.

Engineering Materials Performance during Processing and Service in (Severe) Environments
M. A. J. Somers, DTU Mechanical Engineering; K. Ståhl, DTU Chemistry.

The performance of metallic materials during service is to a large extent determined by the microstructure induced by the processing history. Targeted optimisation of materials performance requires designing and processing the optimal microstructure. This is only possible through a deep understanding of: 1) The relation between materials processing and the resulting microstructure, and 2) the response of the microstructure towards an environmental load of thermal, mechanical or chemical nature.

All manufacturing processes for shaping or joining of materials include (thermo-)mechanical or (thermo-)chemical treatments causing plastic deformation and/or phase transformations. These imply a change of the microstructure by the formation of new grains/phases, elastic straining, plastic straining causing rotation of the crystal lattice, and changes of density and distribution of defects. Subsequent surface engineering treatments add functionality and protection by a further targeted modification of the (local) microstructure through mechanical or chemical processing.

In situ characterization of the evolving microstructure and monitoring its response to heating and cooling, mechanical loading (tension and compression, cyclic loading (fatigue), strain path changes, creep), chemical environments (gaseous, aqueous) and combinations hereof has been a long time wish of materials scientists and engineers.

By imaging of phases, grains and subgrains during loading as a function of time, the following microstructural information is obtained: Spatial distribution of shapes and lattice orientations of grains,[39] inter- and intra-granular stresses and strains,[40] kinetics of nucleation and growth of phases and grains,[41] and thermal and mechanical stability of phases.[42]

Such information on individual features must be complemented by investigations in diffraction mode with higher angular and/or temporal resolution to gain information on: Quantitative texture,[41a] micro/macro stress,[43] line-profile analysis,[44] (fast) transformation kinetics,[41b, 43] and behaviour in complex (severe) testing environments.

This data provides the necessary input for modelling of microstructure stability, lifetime prediction and targeted improvement. To accurately study samples under real conditions sample environments with heating and cooling in various (inert and reactive) atmospheres must be designed. Additionally sample environments for mechanical testing at room temperature and elevated temperature are needed. These sample environments should ideally be designed for use at both end stations to combine recording of fast kinetics with a full characterization of the microstructure at certain times.

Stabilization of new Materials at Extreme Conditions
M. Bremholm, AU Chemistry.

Discovery of new materials has always been a fascinating discipline in materials chemistry and continues to be of key importance for further advancements in materials science. Use of high pressure allows unique opportunities to stabilize new compounds and this is exploited in the first high-pressure laboratory in Denmark. We have established the auxiliary infrastructure required to perform structure-property relations using diamond anvil cells (DACs) using synchrotron PXRD, which is the principal technique for the structural characterization. Synchrotron diffraction experiments at high pressure are a powerful way to obtain a deeper understanding of atomic interactions and are well-suited for comparison with theory.[45] Extreme pressures are conveniently obtained by placing a sample between the faces of two diamonds and this makes it possible to achieve pressures well beyond 1 Mbar. Heating is obtained by resistive coils (25-800^{o}C) or lasers (500-5000^{o}C).

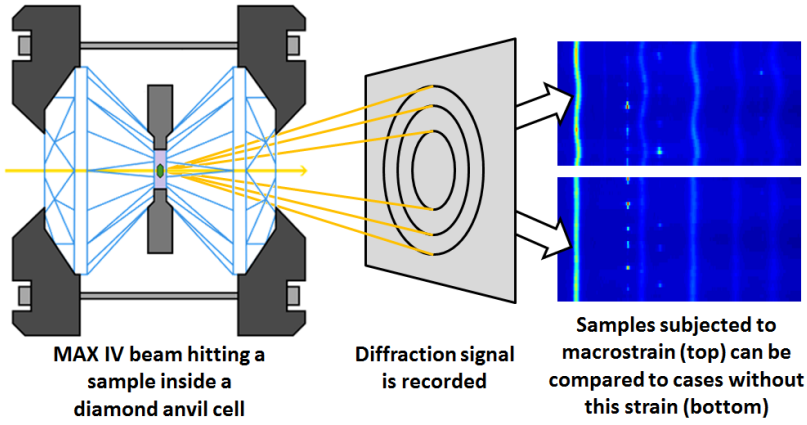

Figure 11: Schematic view of a sample in a diamond anvil cell.

A synchrotron beamline like DANMAX is a prerequisite for *in situ* studies of synthesis at extreme *P,T* conditions and it is furthermore ideal for structural studies at high pressure[45] and this is of particular importance for studies of compounds that are stabilized by pressure.[46] The harder energy-range of DANMAX is ideal for studies using DAC as it allows for high X-ray transmission through the diamonds and a wide range of d-spacings to be probed, despite the limited opening angle of a DAC. The micro-focused beam is perfect for high pressure studies as it allows for laser-heating and targeting Mbar pressures (ideally 3x3 μm^2 beam) while slightly larger beams can be used to improve powder statistics when structural solution and refinement are pursued at less extreme pressure (15x15 μm^2 for $P < 80$ GPa).

Mineral Materials, Geology and Planetary Science
T. Balic-Zunic, KU Natural History Museum.

The study of mineral materials has two primary objectives. As naturally formed solids, minerals hold in their structure the information about the conditions that formed them and are thus basic indicators of geological and planetary processes. On the other hand, due to the inexhaustible variations in natural processes, the resulting richness of mineral appearances presents an everlasting inspiration for material science. As an example of the high-resolution study of complex mineral intergrowths, our recently published study of perthitic feldspars can be mentioned.[47] The most abundant mineral group in the Earth's Crust, feldspars are characterized by complex intergrowths of several phases, which are exsolutions from originally homogeneous mineral phase, developed during the cooling history of the rocks. The study, which combines high-resolution imaging and diffraction with accurate crystal structure characterization of all components included development of new software for the treatment of diffraction data.

The second line of work of the Danish mineralogical research is *in situ* diffraction study of minerals under their natural conditions of formation, which in most cases cover high pressures and temperatures. Although the crystal structures of most of the minerals under surface conditions are known, the mineralogical record of the crystal structure behaviour at the deep Earth conditions is scarce and we are still lacking important information pertaining to the processes that shape the Earth and other planetary bodies. Here, the interest of mineralogists overlaps with the study of materials at extreme conditions in Materials Science. What kind of surprising discoveries of behaviour under non-ambient pressure can be expected from mineralogical studies is illustrated by

the discovery of novel extreme phase transitions in a specific mineral group under high pressures revealed by synchrotron study using DAC.[48]

The development of a combined imaging + PXRD beamline, DANMAX, is a perfect support for this promising area of Geo- and Planetary Sciences.

Bioinspired Materials: Controlling Crystallization by Organic Additives
H. Birkedal, AU Chemistry.

Studies of biological materials have indicated that biominerals are not formed from homogeneous solutions but rather through the formation of an intermediate amorphous phase.[21, 49] This is advantageous e.g. because the amorphous precursor can be molded and shaped into complex shapes prior to crystallization to result in constructs not attainable otherwise. In turn such studies have raised a host of fundamental questions about crystallization mechanisms in general. Bioinspired crystallization takes place at reduced temperature and in water meaning that these approaches intrinsically are 'green' in nature. The use of organic designer additives not only controls nanocrystal growth kinetics, nanocrystal shape and size but also impacts the lattice constants through strain induced by tightly bound organic molecules. This leads to changed properties such as band gaps in semiconductor nanocrystals.[50] The *in situ* X-ray scattering capabilities of DANMAX will be ideally suited for studying such systems that are very challenging because the chemistry takes place in water that has a strong X-ray scattering signal at low concentration. The information obtained will afford insights into growth mechanisms that in turn will facilitate development of improved bioinspired materials.

Environmental Remediation Using Engineered Materials
U. G. Nielsen, SDU Physics, Chemistry and Pharmacy.

The design of materials suitable for strategic remediation of polluted water whether sewage, drinking water or freshwater lakes requires a fundamental understanding of the interaction between the pollutant and the materials used for sequestration on the molecular level. A common strategy is to selectively sequester the pollutants by ion-exchange resulting in structural changes of the ion-exchanged materials or by addition of chemicals to the water resulting in the precipitation of solid phases, which may be detected by PXRD. Characterization of such systems, especially *in situ*, is challenging and requires synchrotron radiation[51] as multiple phases, often of low crystallinity, are present. Conventional PXRD data obtained on in-house diffractometers generally fail to identify minor phases unambiguously[52] and large quantities of amorphous phases (20-50 %) are not detected.[53] Thus, the PXRD results contradict information obtained from other techniques e.g, solid-state NMR spectroscopy,[54] electron microscopy, and EXAFS complicating the analysis. The unique PXRD capabilities at DANMAX will alleviate this gap and allow studies of complex materials. Moreover, the *in situ* capabilities at DANMAX will allow the group to monitor the structural changes upon sequestration and release of pollutants in real time under realistic conditions.[51] This will provide a detailed insight into the mechanism for sequestration of pollutants. The imaging capabilities present unique possibilities for detailed studies of environmental samples such as soils, precipitates and other complex matrices.

Pharmaceutical Materials Science
A. D. Bond & J. Rantanen KU Pharmaceutical Technology and Engineering; T. Rades, KU Pharmaceutical Design and Drug Delivery

In the pharmaceutical industry, it is of vital importance to understand and to control the outcome of crystallization processes and to monitor stability and structural changes in pharmaceutical solids. Our ability to achieve these goals is reliant on progress in pharmaceutical materials science. In this research area, synchrotron X-ray diffraction methods are used to monitor the sequences and kinetics of crystallization from solutions[55] and to follow chemical changes such as hydration/dehydration in crystalline pharmaceuticals under variable temperature or relative humidity conditions.[56]

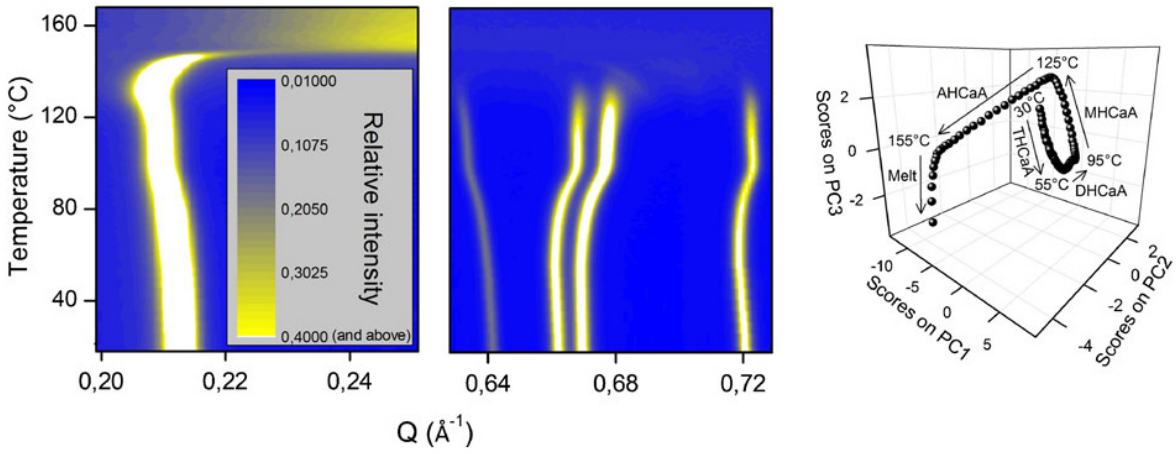

Figure 12: Monitoring dehydration in hydrates of calcium atorvastatin (Lipitor®) by variable-temperature synchrotron PXRD, with a derived principal component analysis (PCA) of the changes that occur.[56a]

The goals of DANMAX – to work on *real* materials studied under *realistic* conditions at *realistic* time scales – are ideally suited to these research goals. The enhanced flux of the X-ray source will enable us (for example) to look more closely at the crucial early stages of crystallization, while the emphasis on sample environments and detectors will allow a wider variety of structural changes to be investigated with significantly improved time resolution. This will provide a tremendous advantage to move forward in this industrially important research area.

Crystal Structure Determination of Molecular Crystal Structures
J. van de Streek, KU Pharmaceutical Technology and Engineering

The group have been solving molecular crystal structures of industrially relevant materials from PXRD data for more than a decade (see e.g. Burley*, et al.* [57] for a pharmaceutical example, ampicillin). Due to the high intensity of synchrotron sources they have even been able to follow solid-state reactions of powders with gases *in situ* at the molecular level.[58] They routinely combine the PXRD data with high-level quantum-mechanical calculations (dispersion corrected density functional theory, DFT-D) to validate the models.[59]

Recently, the group has started research projects in the new research field *NMR Crystallography*, where molecular crystal structures are determined from solid-state NMR data in combination with DFT-D calculations.[60] ss-NMR data are obtained from a sample in powder form, and are therefore

naturally combined with PXRD. The combination of ss-NMR and excellent PXRD capabilities at DANMAX will allow the group to go beyond the standard static description of molecular structures and to determine dynamic behaviour.

Experimental Charge Density Determination from Synchrotron PXRD
J. Als-Nielsen, KU NBI; J. Overgaard & B. B. Iversen, AU Chemistry.

The charge density of a molecular-scale system is the most information-rich observable available in natural science. It can be obtained either from quantum mechanical calculations or estimated experimentally from accurate X-ray diffraction data. We have for two decades invested very strong efforts in measurements and analysis of X-ray CDs on a wide range of systems such as metal organic framework systems, thermoelectric materials, zeolites, molecular magnets, oxidation catalysts, organic solids etc. (see e.g. Jørgensen*, et al.* [61] for a recent review). These studies have been done using single crystal X-ray diffraction, which generally is assumed to produce more accurate data than PXRD. This is the case for typical molecular solids, but for high symmetry inorganic crystals containing heavy atoms, there are e.g. significant problems with extinction and absorption effects. We recently showed that accurate structure factors can be obtained from synchrotron PXRD data, and that these are suitable for multipole charge density modelling.[62] This gave us the idea to develop a new all in vacuum diffractometer, which provides the ultimate accuracy in extracting structure factors.[63] The instrument has been designed, built and commissioned at PETRAIII as a collaboration between University of Copenhagen, Aarhus University, JJ X-ray and DESY. The main aim of the instrument is to reach the fundamental lower limit of background in the data, which corresponds to Compton scattering from the sample. If this is reached the corresponding data will have the best possible signal to noise, and provide the optimum experimental condition for measuring accurate Bragg diffraction data to the highest possible resolution in reciprocal space. A proof of principle study of diamond has been published, and this revealed that the vacuum diffractometer is capable of measuring the most accurate structure factors ever reported.[64] The data have for the first time provided clear experimental evidence for core electron polarization during chemical bonding as previously predicted from theory.[65] The possibility to experimentally probe the effects of chemical bonding on the core electrons opens up a new frontier in chemical bonding studies. One of the critical features in these studies is to limit the peak overlap, and the beam characteristics of DANMAX will make it possible to measure the highest quality structure factors in the world.

Structural characterization and catalyst development
L. F. Lundegaard & A. P. Molina, Haldor Topsoe A/S.

Approximately 60% of all chemical products are made using a catalyst. This is because catalysts make it possible to run the chemical processes at industrially feasible conditions of pressure and temperature and make the process more selective towards the desired product. Rational design of catalysts requires a detailed understanding of the catalytic system at different length scales (See Figure 13). Both synchrotron X-ray diffraction and X-ray tomography techniques have a high potential to contribute to a better understanding of our catalysts.

High resolution X-ray diffraction data and pdf analysis provides a detailed atomic structure of the catalyst phases[66] and the high penetration power of hard X-ray makes it possible to study the active phases in situ under industrial relevant conditions. Making use of a small focused beam, spatially resolved studies can follow the evolution of the catalyst phase composition as a function of position in the reactor, and the high intensity beam, resulting in short exposure times, allows us to

observe changes in the catalyst as they happen.

X ray tomography does not only give a 3D visualization of the catalyst structure but also detailed information of its pore system. Diffusion of gases in the 3D pore structure can be modeled and parameters like surface area, total pore volume, pore connectivity (tortuosity), pore size and pore shape can be obtained. Some of these parameters are required to get a realistic picture of the properties at the macroscale.[13a] Catalysts based on supported porous materials can suffer from intraporous diffusional resistances that dramatically lower the catalyst efficiency. A good connectivity between the pores is essential for the catalyst performance. High resolution imaging reaching down to 50nm or below is of utmost interest in this case.

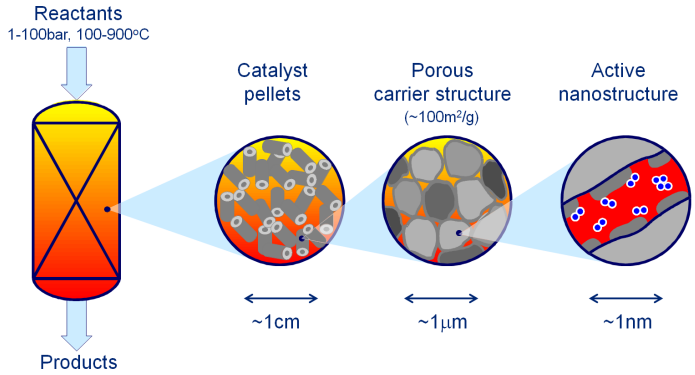

Figure 13: Illustration of the multiple length scales in a catalyst system.

A good strength of the catalyst is also crucial and knowledge on the evolution of grain size and morphology of porous ceramic catalysts during processing and later during operation is of utmost importance in order to design and produce efficient and stable catalysts. X ray tomography can provide unique information to understand the sintering kinetics of the material.

A combination of X ray tomography with diffraction is seen as a powerful multimodal technique to follow aging and deactivation of catalysts in situ under controlled conditions in systems where i) loss of pore volume due to sintering, ii) formation of new phases and/or iii) solid diffusion of active metals and promoters are an issue. Combined with fluorescence detection these approaches would reach its full potential.

It is a great opportunity to have a world-class synchrotron beamline with X-ray diffraction and X-ray tomography within one-hour drive from our location. We are looking forward to make use of DANMAX in the near future for our catalyst development.

Catalytic Nanoparticles from Supercritical Flow Synthesis

L. H. Christensen & H. F. Clausen, Danish Technological Institute, Nano- and Microtechnology.

Catalysts are key components in chemical industry and research. Development of catalytic materials is continuously ongoing to fulfill the increasing environmental and economic demand. In fuel cells, the general goal is to provide catalytic structures with more efficient conversion, in order to achieve fuel cells with greater power output, but also to minimize the use of precious metals.

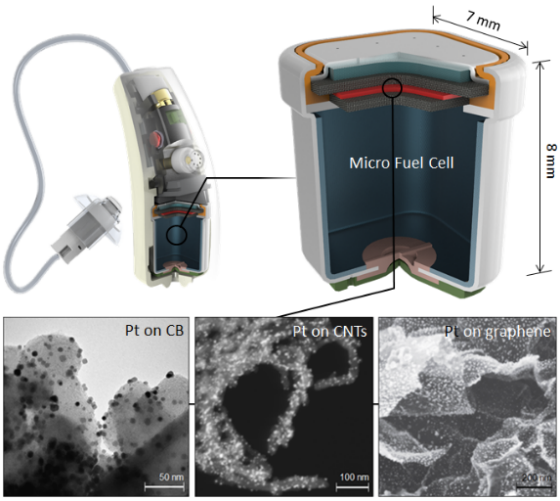

Figure 14: Hearing aid with micro fuel cell. Inserts show electron microscopy images of the catalytic nanoparticles.

Stricter emission regulations are being implemented and generating challenges for the diesel industry by setting higher demands for the removal of pollutants. Improved emission catalyst particles for the removal hydrocarbons, carbon monoxide, and NO_x gasses, are key components for improved emission systems, and cost-effectiveness and conversion efficiency are essential points for industrial success.

The backbone of catalyst development is a fundamental understanding of both how the catalysts are synthesized, as well as how they work during real time conditions. The supercritical flow synthesis (SCFS) is characterized as a green chemical process, and is capable of producing well-defined nanoparticles with re-producible properties. The formation and development of catalytic nanoparticles in the supercritical regime can be monitored using *in situ* PXRD and in combination with PDF analysis can provide the needed information to tailor-made catalytic materials.

The green technology of SCFS is coming on age and at the Danish Technological Institute a fully automated pilot production facility is under construction, which will be able to produce up to 1 tons of nanoparticles per year. SR-PXRD provides the ultimate source for investigating the final synthesis parameters, as minor impurity fraction of the samples becomes visible in the bright beam. The analysis of SR-PXRD data can also reveal the size and size distribution of the catalyst product, which are parameters governing the final properties of the material. These investigations of real catalytic materials at DANMAX could ensure that state of the art catalysts will be developed and supplied for the industry.

Thermoelectric Modules under Operating Conditions
H. Yin, TEGnology APS.

Thermoelectric efficiency depends on the figure of merit, zT. A high zT value is a prerequisite for high conversion efficiency from heat to electricity. High zT materials are usually crystalline semiconductors or intermetallic materials with complex unit cell structures.[67] New synthesis routes and modern fabrication methods pave new paths for structural engineering of TE materials.[68] PXRD is the most powerful tool in terms of crystal structure analysis.

The application of thermoelectric devices often involves harsh environment, such as high temperature, temperature gradients, corrosive ambiance and mechanical force, not to mention that barrier layers, electrodes, possibly protective coatings attached to the thermoelectric material. Thus, the stability of the materials needs to be investigated under these conditions. The combination of real time imaging and PXRD of real samples in real conditions will provide crucial knowledge on the evolution of the grains as function of temperature and/or chemical potential, as well as the thermal expansion mismatch on the interfaces. This in turn will provide valuable information for improving device performance and optimizing manufacture processes.

5. Industrial Collaborations

The interaction with industry is foreseen to take place primarily through two portals, an imaging industry portal at DTU and a diffraction industry portal at AU. The portals will have local equipment for trial runs and less technically ambitious tasks.

The imaging industry portal was inaugurated January 2014. At the time of writing it comprises three local instruments and a staff of 5 people. 18 industries have used the facility so far. The diffraction industry portal is currently transitioning from the planning phase to an operational phase. Initial contact to several companies has shown that there is a great demand for an efficient collaboration between industry and university research.

6. International Status, Potential Local and International Collaborations

6.1. International Status: Imaging

3D and 4D X-ray imaging has been one of the major success stories of third generation synchrotrons. Imaging beamlines are typically heavily over-subscribed, and publication rates for research involving X-ray tomography are very high and growing: currently the level is over 2000/year.

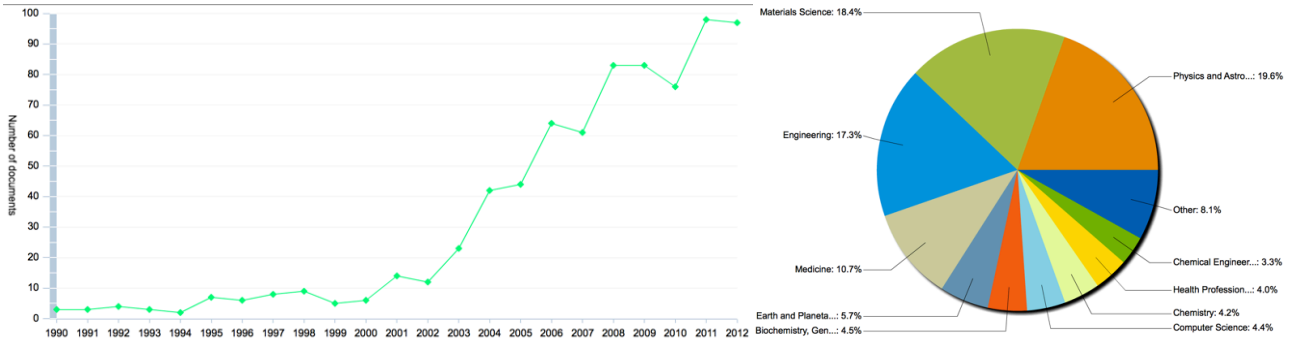

Figure 15: Publications in physical sciences involving "4D" or "*in situ*" applications of X-ray tomography in the period 1990-2012, as a function of year (left) and research discipline (right). Courtesy of S. Hall 22/10/2013.

The rise in applications of 4D and *in situ* X-ray imaging is very significant over the past 10+ years, as shown by Figure 15. This growth is facilitated both by advances in beamline technology and in sample environment options and is set to continue. The main research applications of 4D and *in situ* imaging are material science, engineering and physics (Figure 15 right), which are also key target areas of DANMAX. Diffraction contrast imaging techniques (3DXRD and 3D-DCT) have emerged and matured, within the past 12 years and are exhibiting a rapid rise in number of applications.

Around the globe many synchrotrons plan to upgrade to become "ultimate storage rings", that is to apply the accelerator technology pioneered by MAX IV. Generally speaking, X-ray imaging and X-ray microscopy are seen as two of the major science drivers.

6.2. International Status: Powder X-ray Diffraction

PXRD is the most powerful and most used method of structural determination for materials science. The technique has been revitalized with the advent of intense synchrotron sources and advanced modelling possible with modern computers, e.g. crystal structure solution from powder X-ray diffraction and modelling of PDFs using Monte Carlo and MD simulations.

Current PXRD beamlines at existing sources are highly oversubscribed (2-4 times for the ID11 instruments at APS and 2.4 times at ID31 at ESRF before the shutdown in late 2013) and among the beamlines yielding most publications (The high throughput beamline, 11BM, at APS yields more than 100 papers a year, ID31 on average 70 papers per 200 beam days).

6.3. Local and International Collaborations

- DANMAX have a strong overlapping science case with the neutron hybrid diffractometer HEIMDAL at ESS. HEIMDAL will facilitate *in situ* studies combining thermal neutron powder diffraction and imaging on the same sample. The high brilliance of ESS and the large detector coverage of HEIMDAL will allow collection of diffraction tomography data. The neutron setup will allow larger sample and bulkier sample environment, which in some cases may be necessary to look at real samples under real conditions. The n/X-ray contrast difference therefore makes DANMAX and HEIMDAL highly complementary in capabilities even though they address a similar science case.
- There is also an evident synergy with the ODIN imaging beamline at ESS. The collaboration currently involves 2 PhD students.

7. Technical Design Suggestions

The technical design is in a rather early stage. This is mainly due to pending discussions on the MAX IV contribution to the budget. This will e.g. determine whether parallel access to the two beamline instruments is possible. Parallel access would evidently double the amount of beamtime available, and is of key interest to both Danish and Swedish users. Prime optics solutions are dependent on whether this is feasible.

In this chapter we therefore present a number of technical solutions but refrain from presenting the overall layout of the beamline.

One overriding guideline in this CDR though has been to keep the beamline sufficiently short to avoid the costs of an extension building and thus to enable investment to be focussed on the undulator and detectors. DANMAX will, with this baseline design, comprise an optics hutch and an experimental hutch hosting two instruments within the perimeter of the ring.

7.1. Undulator

The choice of undulator is currently undecided and will depend on management decisions in relation to machine operation and cost. The conservative and cheap solution is to use the same undulator design used for NANO-MAX, the pmu18p5. This undulator is 1.8 m long and composed of 85 periods with a period length of 18 mm with a maximum K value of 1.95 and a gap range from 4.2 to 40 mm. It enables the X-ray energy range from 9 keV to 35 keV to be reached in a continuous fashion using the 5^{th} to 21^{st} harmonics. Some numbers for energy bandwidth and divergence of the beam are given in Table 1.

Energy	Harmonic	K value	Peak brilliance	DE/E at 1% of max brilliance	s' (h*v) (µrad)
9 keV	5^{th}	1.770	1.3e+21	1.58%	14.6x13.7
20 keV	11^{th}	1.775	2.2e+20	1.21%	14.6x13.7
30 keV	17^{th}	1.800	4.0e+19	1.16%	14.8x13.8
35 keV	21^{st}	1.882	1.8e+19	1.15%	15.1x14.2

Table 1: Beam parameters with the NanoMAX undulator pmu18p5.[1]

A 4 metre long version of the same undulator design is estimated to provide at least a factor of 3 increase in photons on sample; this solution is used for the budget below. Simulations by the company DANFYSIK point to an additional increase by a factor of six by using a liquid Nitrogen cryogenically cooled undulator.[69] These gains in efficiency are considered to be strongly relevant for DANMAX as they imply that the beamline can offer some unique features:

- *Improved phase contrast*. With the better choice of undulator DANMAX will enable operation in a space-time resolution window that is an order of magnitude improvement from state of the art (see the status and evolution of fast tomography shown in Figure 16). The aim is not be to break the record for time-resolved studies (implying also mechanical challenges), but rather to improve contrast and spatial resolution for a fixed duration of the tomogram acquisition.
- *Improved diffraction contrast*. With the superior choice of undulator grain mapping of say 1000 grains these can be accelerated from taking typically a few hours to a few minutes. This will strongly enhance possibilities for both multimodal and multiscale mapping.
- *In situ experiments*. The high flux from the undulator combined with the fast readout of the detectors will allow a superior time resolution.
- The *small divergence* of the undulator radiation, compared to e.g. a wiggler, will allow high resolution PXRD experiments without the need for analyzer crystals. This will greatly speed up the data collection time.

7.2. Imaging: Contrast Mechanisms and Optics

Absorption & phase contrast: The trend in the synchrotron imaging community is towards an almost exclusive use of pink beam (bandwidth of ~2%) operation. Priority will not be on being able to scan near edges, as reliance only on pink beam operation is deemed acceptable.

Diffraction contrast: Experience in the 3DXRD/DCT community points to bandwidths of the order $\Delta E/E = 3x10^{-4}$ to 10^{-3} as optimal. Adaptable primary optics that matches this specification have recently been proposed for X-rays above 30 keV, where combinations of transfocators and Laue crystals can be used. However, using such a solution with the energy range of interest here is a challenge. Conventional Bragg-Bragg monochromators is the default solution.

Dark field contrast using gratings: This mode does not pose additional constraints on the primary optics.

Based on this, two alternative strategies for the primary optics have been identified, as described below.

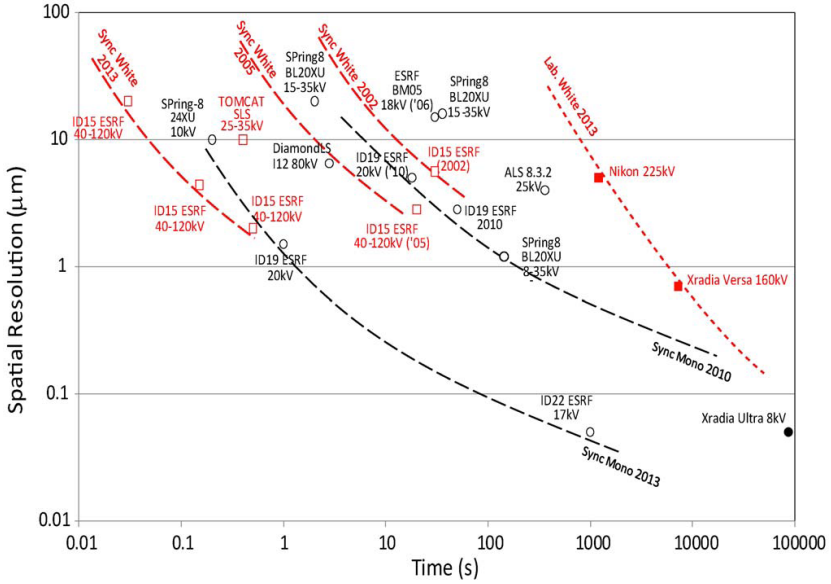

Figure 16: Evolution in fast X-ray tomography at synchrotrons and lab sources.[70]

- Multilayers: Two multilayers in Bragg-Bragg configuration are well adapted to define and transport a pink beam. We propose adding a transfocator for minor adjustments of beam size. A conventional Bragg-Bragg crystal solution can be incorporated in a cost-effective way by depositing the multilayer on Si crystals and leaving a stripe with no deposit. The multilayer solution is common at other tomography beamlines and MAX IV will have prior experience with cooling and other operational issues.

The main disadvantage of this solution is that multilayers are known to degrade the coherence properties of a beam. The ESRF-ID19 staff has partly overcome this by design optimisation and software corrections.

- Use of CRL to form a secondary source: Using a transfocator and a cooled pinhole, a virtual source can be made that singles out one individual undulator peak.[71] In this case, a Bragg-Bragg monochromator has to be added for the diffraction contrast mode of operation. A disadvantage of this option is that the beamline would preferably be longer.

7.3. Powder X-ray Diffraction: Optics, Instrument and Sample Environments

For high resolution PXRD a conventional Bragg-Bragg monochromator is needed to maintain a good peak resolution. To obtain the best possible resolution we propose to use beryllium lenses to obtain a truly parallel beam. The focal length of such lenses is dependent on the photon energy and the number of lenses has to be varied with photon energy. The beryllium lenses will deteriorate the coherent properties of the beam, however, this may be an advantage for PXRD.
An interesting option is a high flux mode where a wide energy band is transported to the sample using multilayers as described above. This option would lead to an overall lower instrumental resolution but a better time resolution. The high resolution mode (with Bragg-Bragg mono-chromator) will be the standard mode of operation.
The beam size for PXRD should be around 100µm to ensure a good powder average. Smaller beams are necessary for high pressure cells and for diffraction tomography (down to approx. 1µm FWHM). Beam focusing will be done using compound refractive lens.

Detectors: The development of X-ray detectors is presently evolving at an incredible pace. Commonly used CCD or CMOS technologies are rapidly being displaced by hybrid pixel detectors due to their high frame rates, very high dynamic range, and low (often negligible) noise levels. As the technology is rapidly evolving and maturing the procurement of the detector systems will not be commenced until the end of the construction to ensure that the systems are state of the art. We are proposing the following detector types:

- *2D: Dectris Eiger X 16M with thick Si conversion layer or equivalent.*
  The small pixel size of the Eiger X detector is highly attractive feature especially in the light of the near parallel beam at MAX IV. However, the thin Si conversion layer in the current generation (450µm) leads to a lower overall efficiency of the detector, especially at high photon energies. The Pilatus X series have a disadvantage of larger pixels, however, it is available with a 1000µm Si conversion layer and will have a factor of ~2 higher efficiency at higher photon energies.
- *1D: Dectris Mythen 24K or equivalent.*
  The Mythen system can be extended to a system covering 120°. The detector will have small gaps between the module so a sturdy goniometer is needed to shift the detector slightly to obtain full coverage in two settings. This movement needs to be fast, reliable and precise to facilitate *in situ* experiments.
- *Energy dispersive detector: Canberra Si(Li) or equivalent.*
  This detector will be used for fluorescent elemental mapping in combination with position resolved PXRD. This type of detector does require liquid nitrogen cooling.

Sample environments: The focus of DANMAX is *in situ/operando* experiments and therefore many different sample environments are an essential prerequisite. The sample environments will be developed in parallel with construction and commissioning of the beam line. Development will be performed as collaborations between the beam line staff and the users in the DANMAX consortium. After the construction and commissioning period we plan to have a strong suite of available 'standard' sample environments, e.g. mechanical loading, gas loading, furnaces, cryostats, solvothermal conditions, battery charge/discharge, diamond anvil cells etc.

General design considerations: The two key components of the end station are the *sample environments* and the *detectors*. The goniometer will be designed around these to give maximum flexibility to add custom-made sample environments. Additionally the detectors will be able to

move completely out of the beam to facilitate the installation of complete custom build equipment, e.g. the all-vacuum diffractometer described by Straasø, *et al.* [63].
Additionally, it is important to consider the infrastructure right from the design phase. The beamline must support chemistry experiments, i.e. access to chemistry laboratories should be convenient, gas manifolds should be available from day one, and sufficient ventilation must be available in the hutch. Additionally the hutch should be designed in a way that satisfy local rules and regulation on laser safety to allow users to bring sample environments incorporating lasers e.g. for heating or optical spectroscopy.

8. Management

The framework for the management and operation of DANMAX will be formulated in a contract between MAX IV and DTU, on behalf of the DANMAX consortium. This is foreseen to be supplemented by a collaboration contract between the Danish stakeholders. The DANMAX consortium will elect a board. Provisional members are:

- Henning Friis Poulsen, DTU
- Bo Brummerstedt Iversen, AU
- Robert Feidenhans'l, KU
- NN, representing other universities: AAU, SDU and RUC
- NN, representing private sector interests
- NN, MAX IV

Private sector use: Access to DANMAX will primarily be through the two industry portals. The aim is provide a comprehensive service to interested users that do not have the background required for gathering and processing data on their own. By pooling tasks, a much reduced turn-around time can be offered. Tasks will typically also include the use of local X-ray equipment (at DTU and AU) for preparatory studies and for the less demanding jobs. Financing will typically be arranged on commercial terms or involve research collaboration between industry, universities and DANMAX.

9. Budget and Timeline

9.1. Tentative Budget for the Construction Phase

Several key pieces of instrumentation are currently being discussed. Among these are:

- Undulator: A cryo-cooled undulator could provide a gain of six over a hybrid undulator solution (see above) but is considerably more expensive. We have an offer for 14 M DKK.
- Two independent stations: This option will be moderately more expensive.

A tentative budget (using the 4 m long version of the conventional pmu18p5 undulation) is included in Table 2:

	M DKK	
Personnel: design and construction	**16**	
Equipment	**89**	
Undulator		10
Frontend		5
Optics and beam definition		20
Imaging end station		18
Powder X-ray diffraction end station		14
Sample environments		3
Infrastructure		19
Software development	**5**	
Total	**110 M DKK (= 134 M SEK)**	

Table 2: Tentative budget for the construction phase. DKK/SEK ~1.22

We anticipate the beamline to be financed as follows:

	M DKK	M€
NUFI, Danish research infrastructure fond	35	4
Danish universities	25	
Danish regions	20	
MAX IV	30	
Total	**110**	

Table 3: Beamline financing

9.2. Budget for Operational Phase

The Danish stakeholders will finance the staff of the beamline, defined as 2 senior scientists, 2 postdocs and 1 person year technical staff equivalent. MAX IV will cover other operation costs. The staff will be employed by Danish universities and stationed in Lund. They will have a dual affiliation between their employing university and MAX IV.

The commercial income will be shared between the two industry portals, DANMAX and MAX IV. We foresee that part of this income will be set aside for future upgrades of the beamline.

9.3. Timeline

Construction phase: 1/1 2015 – 1/7 2018
Commissioning phase: 1/7 2018 – 31/12 2018
Operational phase: 1/1 2019 –

Milestones (with indication of number of months after start of project):

1. Appointment of DANMAX responsible (month 3)
2. Two day workshop for consortium and other stake holders (month 4)
3. Completed CDR, subject to external review (month 6).
4. TDR, subject to external review. Will be developed in close collaboration with MAX IV (month 24).
5. Financial auditing (month 30)
6. First ”DANMAX yearly meeting” (month 30)
7. External users (month 48)

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Appendix 1. Partners in the DANMAX Consortium

University partners:
AU, Chemistry: Professor Bo Brummerstedt Iversen, Associate professor Torben R. Jensen, Associate professor Henrik Birkedal, Associate professor Jens Erik Jørgensen, Associate professor Mogens Christensen, Senior researcher Jakob Overgaard, Associate professor Jørgen Skibsted, Assistant professor Martin Bremholm, Assistant professor Nina Lock
AU, iNANO: Associate professor Jeppe Vang Lauritsen.
AU, Engineering: Associate professor Jens Vinge Nygaard
AU, Acroecology: Professor Lis Wollesen de Jonge
DTU Physics: Professor Henning Friis Poulsen, Senior researcher Søren Schmidt, Research engineer Carsten Gundlach, Research engineer Jette Oddershede
DTU Energy Conversion: Head of section Luise Theil-Kuhn, Senior researcher Poul Norby, Senior researcher Jens Wenzel Andreasen, Senior researcher Jacob Bowen.
DTU Mechanics: Professor Marcel Somers, Associate professor Wolfgang Pantleon
DTU Wind: Professor Dorte Juul Jensen, Professor Bent F. Sørensen, Senior researcher Søren Fæster
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