Draft for a beamline on:
Non Crystalline Diffraction with Microfocus for Life and Materials Science on ALBA

"The ideal, universal SAXS instrument is one which provides a tunable, narrow bandpass, high flux on a small sample and obviates the need to correct the data for any distortion"
M.H.J. Koch; Makromol. Chem., Maromol. Symp., 15,79 (1988)

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1. The background

Simultaneous small and wide angle X-ray diffraction (SAXS/WAXS) techniques allows one to investigate at different length scales the structure and dynamics of a wide range of systems on interest in medicine, biology and materials processing among others. The simultaneous SAXS/WAXS techniques are one of the most frequently requested methods for studying structural and morphological changes in real time. The principle of this combined method is simple. During the experiment, two position-sensitive detectors are placed in different locations covering a wide angular range such as ~ 100 µrad < θ < 0.5 rad where 2θ is the scattering angle. This means that ca. four orders of magnitude in q (= $4\pi/\lambda$)sin(θ /2)) are accessible. While WAXS provides information about the molecular and atomic ordering of the material, SAXS is sensitive to heterogeneities in the electron density on a larger mesoscopic scale (10-10^3 Å). Nowadays, a step forward in this context aims to obtain such an information with real spatial resolution down to micron and nanometers. Beam sizes of several tens of μm or even sub-μm beams can have a strong impact on fields such as micro and nanotechnology, materials science and life sciences.

In Spain there is a very active and stabilized community on non-crystalline diffraction with synchrotron light. The research effort on the topic is reflected by the relative great amount of experiments carried out in the different synchrotron facilities around the world. Part of this activity is reflected in the Scientific Cases presented below which represent a snap-shot of the state-of-the art in the field.

The proposed beamline is expected to be a high resolution and high brightness beam provided by an vacuum undulator insertion device at CELLS. The beamline should deliver a beam of some tens of μm in diameter in a energy range optimized around 2.4 keV. A modular microfous optics will provide beam sizes in the μm m and sub-μm range. Small and wide angle cameras with associated linear and area detectors for static and time resolved measurements would record the scattered radiation from samples under different and well defined environments. Its modular arrangement will allow a choice between ultra small angle scattering for large fibrous structures or microfocus illumination with simultaneous SAXS/WAXS option for materials studies. At present, there is only one beamline (ID13 at the ESRF) with similar characteristics, being one of the two most demanded beamlines at the ESRF lacking capacity to take up the necessities of the scientific community.

2. The scientific Case

2.1 Biological systems

Time-resolved X-ray fibre diffraction study on "live" muscle tissues

Time resolved X-ray diffraction studies on biological fibrous systems in conjunction with synchrotron radiation provide essential information on the structure and dynamics of large molecular assemblies in low order environment at time-scales ranging from milli-seconds to seconds. Over the past 25 years our understanding of the structure and function relationship in skeletal muscle tissue flows directly from the use of these techniques [1]. Recent advances in crystallography have revealed the atomic structures of the muscle proteins, myosin heads and actin, that when assembled in the native tissue are responsible for muscle contraction [2, 3]. Moreover, the physiological function of muscle at the level of isolated tissue and single cells have been characterised in detail *in vivo* [4, 5]. However, our full understanding of the molecular basis for muscle contraction still remains largely undetermined.

The muscle unit cell is very large, measuring in normal conditions more than 2300 nm, and muscle tissues are weakly diffracting. Consequently, this important biological system can only be successfully investigated with a third generation synchrotron radiation source such as the Spanish source CEELLS. To make significant advances to our understanding of its molecular structure access to an undulator beam line dedicated to SAXS/WAXS type experiments is necessary. The outlined experimental station would permit to record with high spatial and time resolution changes to the molecular ordering in space of the muscle proteins. Ultimately, the long-term aim is to develop a molecular model that can explain the mechanical properties of muscle tissues and its ability to carry out these functions through the conversion of free energy gained from the hydrolysis of ATP.

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Structure and lipid organization of cutaneous tissues

The outermost layer of the mammalian epidermis, the stratum corneum (SC), consists of thin keratinized cells (corneocytes) embedded in a lipid-enriched intercellular matrix organized in lamellae. The main function of the SC, the barrier function, depends strongly on the specific structure of this tissue [1]. Particularly, the barrier permeability is located in the lipid lamellar structure that mainly consists of ceramides, free fatty acids, cholesterol and cholesterol sulfate [2]. A number of skin studies are based on microscopy techniques [3]. However, some aspects related to the influence of the lipid composition on the lipid lamellar organization require techniques that offer a more detailed information and in which no sample manipulation take place.

Some studies about the lipid organization were carried out by X-ray scattering using low energy sources, however the long time exposition of the sample to the X-ray beam made hardly appropriate the technique [4]. More successful results have been published by use of SAXS and WAXS using synchrotron radiation. These studies have demonstrated the SC lipids are organized in two coexisting crystalline lamellar phases: the short periodicity of approximately 6 nm and the long periodicity phase with a periodicity of approximately 13 nm. The 13 nm lamellar phase and its predominantly orthorhombic lipid packing are considered to be crucial for the skin barrier function and their presence is strongly dependent on the lipid composition [5,6]. Recently a controversy have been opened about the correct description of this lamellar phase. This question, and the specific role of each lipid in the lamellar structure and consequently in the functionality of the SC must be still clarified. These facts are probably related with the presence of microdomains in the lipid structure that could be resolved by simultaneous use of SAXS and WAXS microdiffraction. The use of a microfocus beam is also necessary to gain correct information on the structure that in those systems with the available size spot can only be conjectured if it corresponds to a mixture of structures or to separate domains. In fact synchrotron x-ray microdiffraction has resulted very appropriated to study similar systems [7], the reduction of the focus size in the proposed microfocus beam could report even more interesting results in the field of the biological nanostructured systems.

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Interfacial transport phenomena

Equilibrium properties of surfactant system mixtures are, probably, quite well understood at present. In contrast, there is a lack of knowledge on non-equilibrium processes and in the kinetics of phase transformation, particularly in phenomena that take place at an interfacial level when different systems are mixed. These processes play an important role in the properties of thermodynamically unstable systems such as emulsions and vesicles or liposomes and in their preparation methods [1]. Understanding the processes of emulsification can allow for a better control of their properties. Low energy emulsification methods have attracted increasing interest, not only because of energy savings, but also as a way to control their properties (e.g. particle size and stability) [2]. Closely related to the emulsification process is the liposome solubilization by surfactants and the reconstitution of these vesicles by either surfactant removal or dilution [3]. Liposome solubilization by surfactants is important because their application as a simplified model of biological membranes and delivery systems [4] and the reconstitution process is a useful method to insert proteins in lipid bilayers [5]. Thus, the knowledge of the interfacial transport phenomena at the initial steps and the transient states during these processes can give light about the conditions in which these controlled emulsification processes are successful or fail.

In this sense, the construction of a beam line specialised in micro focus scattering-diffraction would be useful to perform experiments in which two liquids are contacted. This would allow to follow the kinetics of structural transitions at the moment and at the concentration-gradients present in real systems. The information gained up to now in this type of processes rely on stopped flow techniques, where only a mean after mixing can be achieved [6, 7]. Also experiments in which a solid is contacted by a liquid could give information on the dynamics of solubilisation of materials deposited on the substrate. The need of the micro focus stands both on the distance resolution of the experiment and on the possible inhomogeneities that can be established in the systems.

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Condensed chromatin within metaphase chromosomes

DNA is packaged in the cell nucleus bound to histone proteins forming a complex supramolecular structure called chromatin. At present we only know the molecular structure of the nucleosome, the fundamental subunit of chromatin. The nucleosome is a cylindrical body of ~11 nm diameter and ~6 nm high that contains 160 bp of DNA in its periphery. The maximum degree of DNA compaction is produced in the chromatids (~600 nm in diameter) of metaphase chromosomes during mitosis [1]. Condensed chromosomes are responsible of the transfer of genetic information to the daughter cells but at present we only have poorly defined models about their structure. The molecular architecture of condensed chromatin inside metaphase chromosomes is completely unknown [2].

The local concentration of DNA in metaphase chromosomes is high [3]. However, most of our knowledge of chromatin structure is based on experimental studies that have been carried out in different laboratories using conditions that produce chromatin fibers having a low local concentration of DNA [4]. Thus, an important long-term research objective is the study of the structure of condensed chromatin with a high DNA density in metaphase chromosomes.

It has been found previously that small chromatin fragments containing from 5 to 35 nucleosomes form very compact cylindrical structures of 30-40 nm diameter [5-8]. In more recent investigations it has been found that these cylindrical bodies aggregate spontaneously and form high molecular mass structures [9] that are similar to the structures seen in the periphery of partially denatured metaphase chromosomes [10].

The structural study of these high molecular mass aggregates and of the different higher order chromatin structures of metaphase chromosomes will require the use of small angle X-ray diffraction. The research group involved in this study has previous experience in the use of this technique in the Daresbury Laboratory for the structural analysis of histone-DNA complexes of low molecular mass [11] and of protein detergent complexes [12, 13].

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Microfocus approximation to single particle scattering/diffraction

The use of a micro focus beam coupled with small angle and wide angle X-ray detection with a high time resolution can allow for the investigation of isolated particles. The usual way to investigate isolated particles involves high dilution of the system to eliminate the interparticle interference contribution [1]. However, not always it is possible to achieve a high dilution without changing the structure of the system. The use of micro focus can allow to investigate the form factor of single particles. The investigation of single particles has also the advantage of the resolution of structures without the always present size polidispersity that always obscure the form factor information. If the time resolution is not good enough, immobilisation techniques of the particles, either on solid substrate or by increasing the medium viscosity, would be needed. The systems where this technique would be applicable correspond to any field of the colloid science. In particular, information about the mechanism of liposome or emulsion solubilisation could be improved [2]. Droplet flocculation and coalescence could also be studied in detail.

The ideal beam size for the type of experiments proposed here would be as small as possible but, getting to sub-micrometer beam size, would already be useful for single particle scattering-diffraction.

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Amyloid fibril formation : biophysical studies

Amyloids are proteinaceous aggregates predominantly formed by β-structures which are spatially organized as insoluble fibrils. Such fibrils have been identified as the main structural components of neural plaques, the deposits found in the Central Nervous Systems of patients with Alzheimer, spongiform encephalopaties and Huntington's disease, to quote just three of a number of diseases associated to the presence of amyloids. In the case of Alzheimer's disease the peptides involved in the formation of the neural plaques are the so called β-amyloid peptides (40-42 residues long) whereas prions are the proteins associated to the development of spongiform encephalopaties. The degree of primary homology between β-amyloid peptides and prions is very low. The similarities of the amyloid structures that both types of proteins can form, however, has brought up the hypothesis of the existence of a common mechanism of fibril formation. In all cases it seems that fibril formation is always preceded by a conformational change which implies the conversion of some α-helical part of the peptide or the protein into a β-structure. From that conversion on, it is believed that amyloid fibrilization evolves via a nucleation process, but the structure of the nucleous (or protofibrils) and of fibrils is not yet known in detail.

On the other hand it is of particular interest the influence that two important elements found in the neural plaques in vivo, cell membranes and glycosaminoglycans (GAGs), have on the alpha-beta conformational transition and in the formation of fibrils. Moreover, recent studies provide evidence that shows that the interaction of amyloids with cell membranes may take place at specific membrane microdomains, the so-called 'rafts', rich in cholesterol and sphingolipids [1]. These lipidic microregions would have a diameter of the order of tens of nm.

It would be therefore advantageous to be able to use using non-crystalline material X-ray diffraction and scattering techniques in order to obtain information on the influence on the structure of the different kind of peptide aggregates (nucleus, protofibrils, fibrils) that amyloidogenic peptides form. Simultaneous use of SAXS and WAXS microdiffraction could be of particular interest for a detailed characterization of the complexes amyloid-'rafts', when biological membranes are present in the system. The feasibility of applying X-ray techniques to the study of amyloid fibrils has already been established [2].

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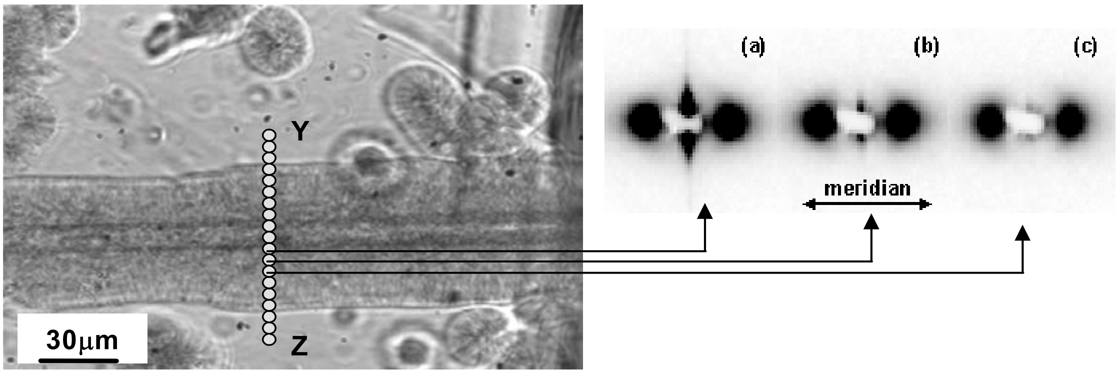
2.2 Polymer science

Polymer Crystallization

For many years nucleation and growth as a stepwise process has dominated discussions about polymer crystallization under quiescent conditions [1]. In contrast to this view a multi-stage process [2] or a spinodal-assisted crystallization process [3-5] has been recently proposed. These ideas have in common that crystallization of polymers is preceded by ordered precursors. In recent years this point has been subjected to an important and still open debate [6, 7]. In the case of shear-induced crystallization so-called shish-kebab structures occur, in which oriented molecules serve as precursor of primary nucleation and form the shish [8, 9]. Further knowledge about the mechanism of the early stages of shear-induced crystallization is of great importance not only for our fundamental understanding of polymer crystallization, but also for the industrial processing of polymers.

The structural and morphological evolution of shear-induced crystallization precursors can be spatially resolved by simultaneous small- and wide-angle X-ray microdiffraction [10]. The sample can be scanned through the beam along a line with μm-range steps (Fig.). A small X-ray beam divergence (less than about 0.2×0.2 mrad^2) allows recording both the WAXS and SAXS signals together in the same pattern [11]. This would allow one to perform innovatively accurate data analysis.



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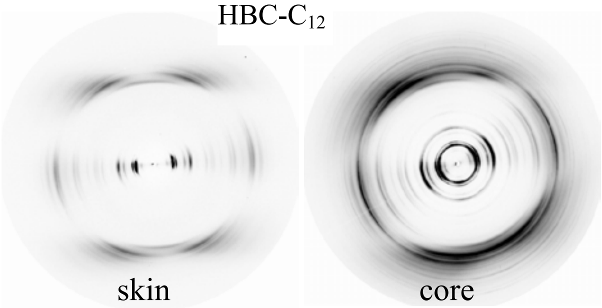
Structure formation in liquid crystalline polymers

A class of organic solids of particular interest is that of liquid crystalline polymers (LCP). The great variety of molecular architectures which are nowadays available offer a rich series of thermotropic behaviors leading to a great variety of mesomorphic structures including main chain[1] and side chain[2]and cholesteric[3] liquid crystalline polymers. The existence in these systems of significant molecular order controls most of the physical properties including rheology, mechanical properties and dynamics among others. Moreover, cholesteric liquid polymers may exhibit interesting sel-associative phenomena of potential interest in biomolecular recognition[3,4]. An special case is that of conducting discotic liquid crystalline (LC) materials. These materials might serve as active electronic components in future devices [5]. In general, the structure, molecular orientation and hence physical properties of liquid crystalline polymers depend strongly on processing, especially such properties related to the anisotropy of the sample. Of fundamental interest id the investigation of skin-core effects on processed samples, like for example, extruded filaments under different processing conditions, as well as the time and spatially resolved ordering developed during the phase transition from the liquid crystalline phase to the solid state during the spinning process and in thermal treatment. Some experiments performed at ID13 (ESRF) with a beam size of 5 µm show some that for conducting discotic liquid crystalline polymer filaments the skin presents a higher degree of orientation than the core[6]:



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Ordering phenomena in confined environments: nanofilms

From a fundamental point of view, the understanding of the development of structures and the reorganization of the polymeric chains in confined environments is challenging. This type of studies becomes relevant when they are directed towards specific problems in applied polymeric materials in which a profound knowledge of these structural features can serve as a tool to control the properties and performance of these systems. In this regard, thin film polymeric materials are of considerable interest due to the possibility of inducing specific properties at surfaces [1,2]. Multilayer systems combine different materials with control layer sequences in order to attain or improve a particular property [3]. Moreover, the possibility of including polymeric chains in crystalline channel structures in which the diameter of the cavities are of the order of nm has been demonstrated and a significant reorganization of the structures and morphologies of these polymers is observed after the confinement in nanochannels [4,5].

Small and wide angle X-ray microdiffraction experiments performed simultaneously will allow to obtain detailed structural information on this type of materials, with the possibility to focus in particular areas with µm resolution, and to determine the differences in morphology and structure induced at the surfaces, or between layers, or after confinement in nano-channels [6,7].

It has been shown that the combination of grazing-incidence small-angle X-ray scattering with a micrometer-sized X-ray beam (µGISAXS) is a powerful thin-film characterization method and allows to gain two orders of magnitude in spatial resolution compared to conventional GISAXS experiments [9, 10].

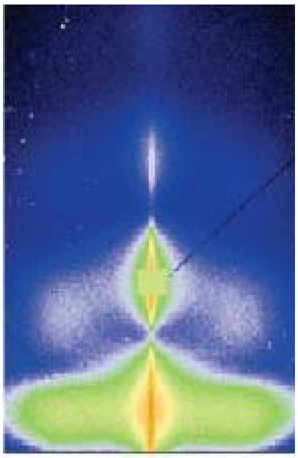

Fig. μGISAXS pattern of a self-assembled nanometer-sized gold cluster layer on top of thin polymer layer on a silicon substrate[10].

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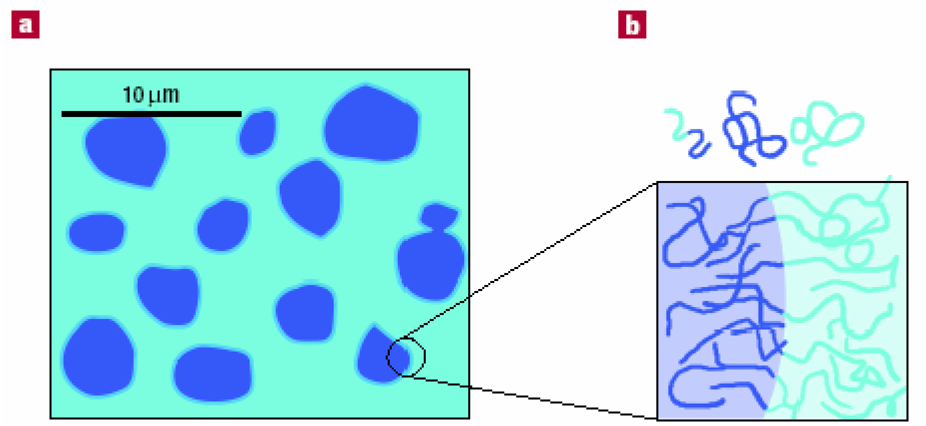
2.3 Multi-component materials

Polymer blends

One of the main areas in the design and development of new polymeric materials with outstanding properties is that of multi-component systems. This kind of materials include polymer blends, copolymers and composites with the incorporation, very recently, of innovative components such as nanofillers or carbon nanotubes as reinforcements. The final performance of the resulting systems depend not only on composition and processing conditions but also on the existence of interactions or phase separation between the components, and on the physical state of each component at the temperature of application.



Adding a compatibilizing agent, such as a diblock copolymer, to a polymer blend can improve its stability. **a**, A two-dimensional slice through a compatibilized blend with a dispersed phase morphology containing a minority dark blue phase and a majority green phase. **b**, A molecular cartoon showing how the diblock copolymer (top left) is segregated at the interface between the two phases.
1. Ref

Microstructure of interphases

The incorporation of reinforcements, fillers or other polymers to a crystallizable polymer matrix will affect the crystallization behavior of the matrix with the formation, in some cases, of particular morphologies at the interphase which can significantly influence the properties of the materials [1, 3]. The understanding of the role of the different variables in the development of the crystalline structure and morphology at the interphase is very important in order to establish their relevance in material performance and to design new technological applications. The use of simultaneous small and wide angle X ray microdiffraction can be a very valuable tool to study the nature and structure of the interphase in these complex multi-component polymeric materials with the possibility of scanning sections in the order of microns and to establish the influence on it of the different components and of the thermal and mechanical history imposed [4-6].
The synthesis of polymer composites based on reinforcing natural fibres such as cellulose is a field of increasing interest [7]. Plant fibres present the advantage of a low cost and ease of recycling. In addition, the combination of natural based fibres with biodegradable polymers such as thermoplastic starch or poly(lactic acid), offers attractive environmentally friendly materials [8, 9]. The surface of plant fibres is covered with pectin and waxy substances, which should be removed for a good adhesion with polymeric materials. Hence, plant fibres usually undergo a chemical treatment to modify their surface. The effect of the chemical modification on the fibre structure and the composite properties, together with the compatibility between fibre and matrix is nowadays an issue of great concern [10, 11]. Micro-focus X-ray diffraction could be used to elucidate the nature of the natural fibre-matrix interphase. In addition, the structural variations within the micron or sub-micron scale across the fibre diameter could also be distinguished using microdiffraction.

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Block copolymers

Block copolymers[1,2,3] are of great scientific interest due to their self-assembled supra-molecular structures formed under various conditions. In diblock AB copolymers, the well-known phase morphologies include lamellae, double gyroids, cylinders, and spheres Many ordered phases have also been observed in ABC type block copolymers. This self-assembling characteristic of block copolymers make then as potential candidates as templates in nanotechnology[4]. Besides amorphous-amorphous diblock copolymers, liquid crystalline-amorphous diblock copolymers and semicrystalline-amorphous diblock copolymers have obtained substantial attention, because molecular and supra-molecular self-assemblies can form at different length scales. In crystalline-amorphous diblock copolymer systems, there are three factors that determine the final phase and crystalline morphology of these diblock copolymers, i.e. the microphase separation of a diblock copolymer (the order-disorder transition temperature, T_{ODT}), the crystallization of the crystallizable blocks (the crystallization temperature, T_c), and the vitrification of the amorphous blocks (the glass transition temperature, T_g). In this type of copolymers, both unconfined and confined crystallization can be observed. The structural characterization of block copolymers can be adequately assessed by WAXS-SAXS techniques[3]. Morover, the use of a microfocus facility may enable characterization of individual phases as well as interfacial regions.

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Nanocomposites

The development of carbon-nanotube and carbon-nanofibre-reinforced polymer composites not only offers unique opportunities to improve the physical and mechanical properties of a given matrix but also allows the evaluation of the intrinsic properties of the reinforcing nanoscale phase. The use of carbon nanotubes and vapour-grown carbon nanofibres as reinforcements has already been shown to improve the mechanical properties of various polymer matrix systems [1, 2]. Standard polymer processing can often be used satisfactorily for these nanocomposites and does not break down the reinforcement material, an issue commonly encountered in short-fibre-composites, which can limit recyclability. Furthermore, initial studies have indicated that the small size of the nanoscale reinforcement allows an enhancement of the properties of delicate structures such as polymer fibres [3, 4]. The key technical challenges which remain for such carbon-nanotube and nanofibre-reinforced polymers are the achievement of a homogeneous dispersion, good interfacial bonding and a controlled degree of alignment Current approaches towards increasing the orientation of the nanoscale reinforcement within the polymer matrix range from optimisation of the extrusion die to stretching the composite melt to form fibres [3, 4]. In addition, changes in the morphology of semicrystalline thermoplastic polymers due to the presence of carbon nanotubes (CNT) and nanofibres (CNF) have been observed [5]. Processing techniques that lead to oriented polymers can induce different crystallization behaviours, but the effects of carbon nanotubes or nanofibres on such oriented polymer systems, although significant [5], have not yet been fully established. Interactions of the nanoscale reinforcement with the matrix during processing and the resulting effects on overall composite performance need to be considered when attempting to evaluate the intrinsic properties of the reinforcement [6]. The above scientific and technical challenges are therefore aimed at exploiting synchrotron radiation microdiffraction facilities to clarify some of the crucial aspects of such novel nanocomposites. The use of locally resolved μWAXS and μSAXS is expected to allow a more detailed investigation of the polymer microstructure as a function of filler type and loading fraction across the fibre diameter. In addition, the study of the deformation behaviour of such nanocomposites during in-situ X-ray experiments should deliver fundamental insights into the nature of the reinforcement effect of nanotubes and nanofibres.

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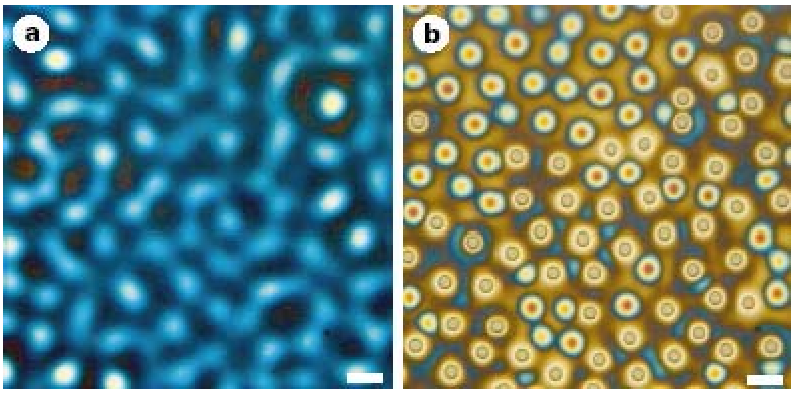
2.4 Polymers under external fields

Electrically induced structure formation

The wavelength of light represents a fundamental technological barrier to the production of increasingly smaller features on integrated circuits. New technologies that allow the replication of patterns on scales less than 100nm need to be developed if increases in computing power are to continue at the present rate. A simple electrostatic technique that creates and replicates lateral structures in polymer films on a submicrometer length scale has been already reported [1, 2]. The method is based on the fact that dielectric media experience a force in an electric field gradient. Strong field gradients can produce forces that overcome the surface tension in thin liquid films, inducing an instability that features a characteristic hexagonal order. In these experiments, pattern formation takes place in polymer films at elevated temperatures, and is fixed by cooling the sample to room temperature. The application of a laterally varying electric field causes the instability to be focused in the direction of the highest electric field. This results in the replication of a topographically structured electrode. Simultaneous scanning µSAXS and µWAXS experiments will be of interest in order to spatially resolve the molecular order induced at different length scales. In addition, *in situ* scanning µSAXS experiments, during application of voltage and temperature and during the subsequent annealing, could be performed in order to follow the different processes of structure formation.



Optical micrographs of polystyrene films which have been exposed to an electric field. In a and b, a 93-nm-thick polystyrene film was annealed for 18 h at 170 8C with an applied voltage U = 50 V. The colours arise from the interference of light, and correspond to the local thickness of the polymer structures.

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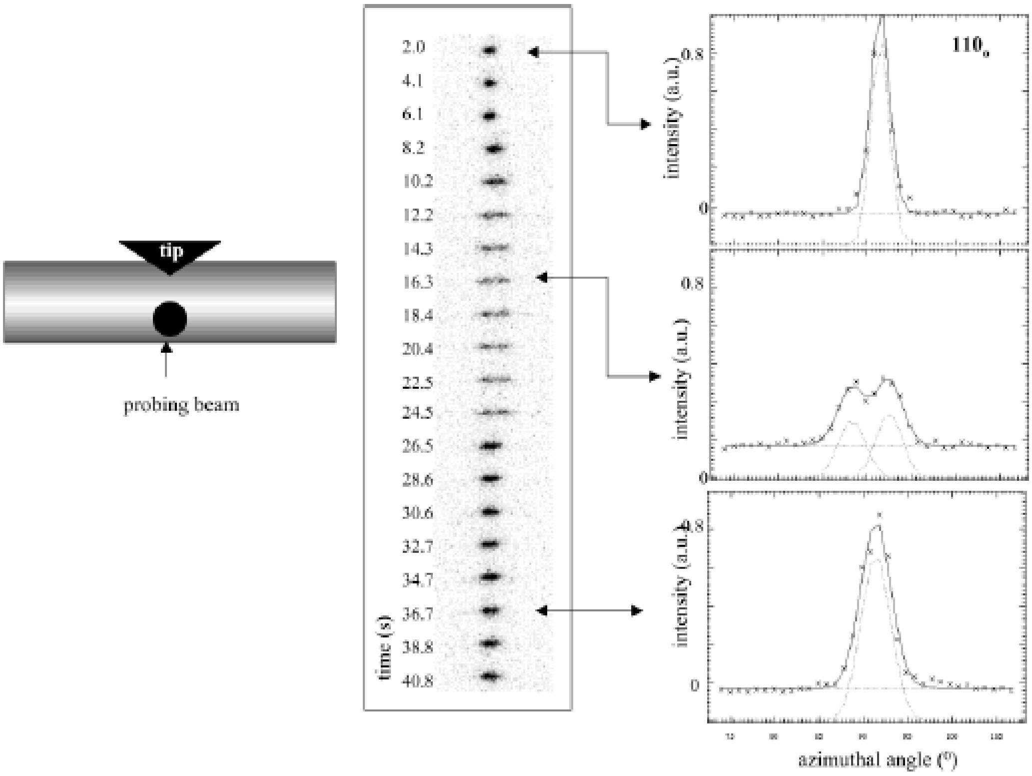
Mechanical Surface Deformation

Indentation hardness offers a convenient way to probe the mechanical properties of a polymer surface [1]. The key advantage of microindentation is the ability to test the material surface in its original assembly, which is of fundamental importance for thin films, coatings, etc. In addition, microhardness offers the possibility to spatially map the mechanical properties in the micron or sub-micron range.

It is now well substantiated, through a number of well established correlations, that hardness of polymers is directly related to the degree of crystallinity and crystal lamellar thickness among other nanostructural parameters [1]. However, there is still very limited information concerning the mechanism of deformation.

Recent simultaneous microindentation and microdiffraction experiments in single polymer fibres using a synchrotron radiation source suggest that the main structural change occurring during indentation is associated with local variations in the crystal orientation, which partially recover upon load release [2, 3]. It has also been shown that plastic deformation may also involve a partial polymorphic transformation [2, 3].



(A) Schematic design of in-situ microindentation experiment on an UHMW-PE fiber. (B) Evolution of 110 orthorhombic reflection during microindentation: at a critical force the 110 reflection splits up into two domains returning to a single domain when the indenter is retracted. Selected azimuthal profiles of the 110 reflection are shown to the right.

However, the above mentioned studies relate to well oriented materials, and there is still no direct information on the mechanism of deformation in isotropic systems. Moreover, the possible nanostructural variations upon indentation such as lattice strains, variations in the size of the crystal blocks, etc, have not been explored in detail so far.

There is a clear tendency nowadays to prepare polymeric materials with enhanced physical properties via a chemical or physical combination of various components at a micro, sub-micro or nanoscopic level. In an analogous way, indentation techniques have progressively developed new instrumentation to probe smaller sample volumes. However, there is still the fundamental need to understand the mechanism of deformation. The local ability of microbeam (or sub-microbeam) techniques to spatially resolve the polymer nanostructure is indeed of great value to provide information on the mechanism of deformation in polymer materials upon indentation.

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2.5 Carbon fibers

Activated carbon fibers: Structure-properties relationship

Activated carbon fibers (ACF) are porous carbons with a fiber shape and a well-defined porous structure which can be prepared with a high adsorption capacity. The main characteristics and advantages of the ACF are the following: i) They have both high apparent surface area and adsorption capacity. ii) They have fiber shape with a small diameter (ranging between 10-40 μ), which are very important characteristics for new applications requiring higher packing density (i.e gas storage). iii) ACF are light materials and can be easily woven into different fabrics (i.e., cloths, felts,…). iv) The pore size distribution of the ACF is narrow and uniform, being essentially microporous materials, although mesoporous ACF can also be prepared and v) The narrow diameter essentially eliminates mass transfer limitations, being the adsorption-desorption rates very rapid.

An issue of special relevance for the characterization of the ACF is their fiber shape, since it introduces differences on the porous texture compared to the conventional AC. In fact, it is not only important to determine the pore volume and pore size distribution of the ACF, but also the distribution of the pores across the fiber diameter, what will be a result of the activation process (i.e., activation temperature, activation method, activating agent) and the precursor used. Although a remarkable effort has been done on the porosity analysis and structural characterization of the ACF, the research on pore distribution within the fibers is scarce and very recent, since it requires a technique with a high spatial resolution. An additional aspect of great relevance is to understand the process of porosity development through in-situ techniques.

In this sense, Small Angle X-ray Scattering (SAXS) technique offers some advantages for the characterization of the porosity in activated carbons, such as it is sensitive to both closed and open porosity. Another important advantage is that SAXS intensity profiles are sensitive to shape and orientation of the scattering objects so that additional information can be obtained in anisotropic studies in oriented samples such as fibers. The spatial resolution needed to accomplish the above objectives can be reached through the use of beams of micrometer size, what results in the micro-SAXS technique. The high intensity of the beams that can be obtained nowadays should also permit to make studies with a sufficiently high temporal resolution, being useful to follow in-situ the reactions involved in porosity development.

Consequently, there exists a link between structure of the precursor and porous texture of the ACF which needs to be well-understood to optimize the preparation. Moreover, it is essential to be aware of a second relevant issue, that is the relationship between porous texture and properties of the ACF, such as adsorption and mechanical properties, as it has been mentioned before.

We have dedicated an important effort to analyze the distribution of porosity across the fiber diameter in isotropic pitch-based carbon fibers and, recently, in anisotropic carbon fibers by using micro-SAXS [1, 2]. These studies have been very useful to understand the differences among preparation methods and precursors. There is still an important piece of work to be done on this area, specially regarding the use of this technique to follow, in-situ and with a spatial resolution of micrometers, the development of porosity during the preparation process.

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3. The requirements

Nowadays there are several beam lines in the world involved in non-crystalline diffraction (ID2, ID13, BM16, BM26 at ESRF; 5-ID, 18-ID at APS; X27C at BNL, A2 at DESY etc). Among them only ID13 and the beam line 11 proposed for DIAMOND are specifically designed to provide microfocus facility.

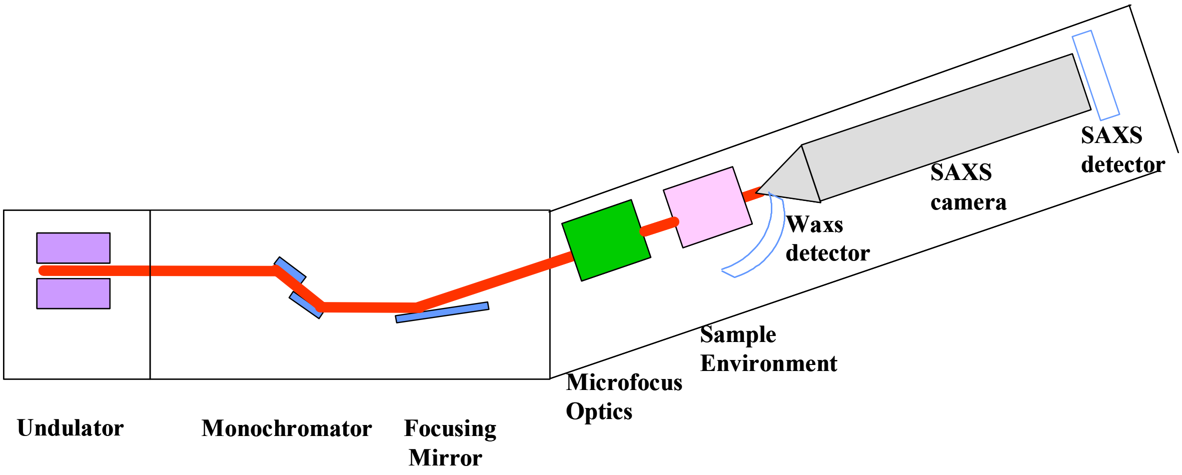
The nature of the users' community with a broad spectrum of research interest ranging from biological to polymer materials suggest the necessity of a beam line at ALBA dedicated to the non-crystalline diffraction with a microfocus facility. The incoming radiation should provide from an undulator device in a low-beta segment of the machine. Microfocus should be provided as a module to be inserted when either illumination of very small samples or spatial resolution were required.

Characteristics of the beam

Undulator: The use of a vacuum undulator will be required due to the higher flux and to the lower divergence in comparison with undulators at atmospheric pressure.
Energy range: Continuous from 0.2 nm (6 keV) through 0.08 nm (16 keV) to 0.07 nm (20 keV)optimized for 0.1 nm (12.4 keV).
Photon flux: 10^{12} Ph/s
Band Pass: 10^{-4}
Beam size at sample/detector: 0.1mm (H), 0.01mm (V) with standard focusing
Divergence at sample/detector: 0.2mrad(H), 0.02 mrad (V) with standard focusing
Beam size at sample/detector: 0.005mm with microfocus pin-hole collimation
Beam size at sample/detector: <0.002 with microfocus set-up based on KB mirrors
Positional stability: 1 % RMS during 1-10 seconds

4. The tentative layout

A scheme of the beam line has been outlined below:



Undulator

- Determination of the characteristics of the undulator

Focusing

- The use of an additional mirror previous to the monochromator in order to collimate and to filter high energy harmonics should be considered.
- A double crystal Si (111) monochromator seems to be adequate (Energy selection and sagittal focusing).
- A focusing mirror either toroidal (vertical and horizontal focusing), or ellipsoidal (vertical focusing) or even a Kirkpatrick-Baez(KB) mirror could be used. Commissioning studies on ID13 indicate that KB mirrors provide ten times more flux for a beam of 0.01 mm with a divergence of about 0.2x0.2 mrad^2

Microfocus

- Pin-hole collimation (0.005 mm)
- Glass capillary optics (0.002mm)
- Kirkpatrick-Baez mirrors (single of two in an orthogonal arrangement) (<0.002 mm). Commissioning studies on ID13 indicate that KB mirrors can provide a beam of about 0.0005 mm with sample to optics distance larger than that obtained by capillary optics.
- Refractive lenses
- Waveguide optics

Sample environment

Sample environment should be adequate to place different systems including :

- Microgoniometer (Microfocus)
- Position scanning set-up (Microfocus)
- Temperature furnaces,
- High pressure cells
- Heating/cooling stage for DSC (Linkam, Mettler etc)
- Heating/cooling shear cell
- Others

The Detectors

Several detectors will be needed in order to cover all the users' demands including:

- Fast area detector with large data storage capacity for time resolved SAXS and WAXS experiments like those related to muscle research
- CCD Area detector for SAXS and WAXS
- Fast linear one dimensional detectors for WAXS (curved arrangement) and SAXS powder diffractometry.

The camera

- The design of the SAXS-WAXS camera should be done to cover a spatial range from 200nm to 0.2 nm.