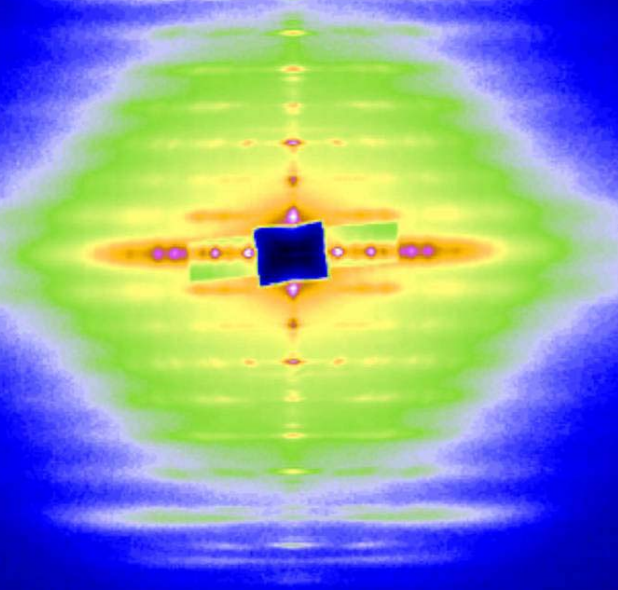
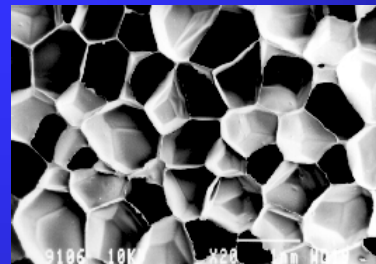
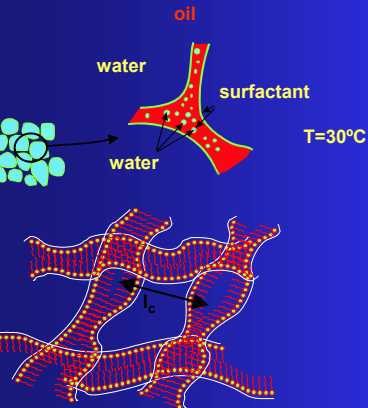
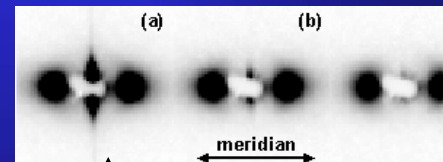
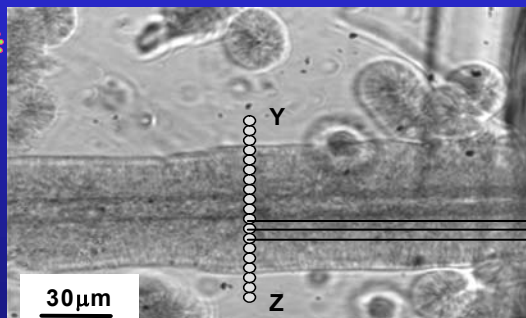
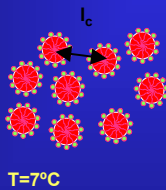


Proposal for a beamline on Non Crystalline Diffraction for Life and Material Sciences with Modular Microfocus Option on ALBA



T=15°C



December 2004

Non Crystalline Diffraction Beamline for Life and Material Sciences with Modular Microfocus Option on ALBA

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Acknowledgements

We sincerely thank Dr. Christian Rieckel and Dr. Wim Bras from ESRF, France and Dr. Igors Sics from NSLS (BNL), USA for useful comments.

Non Crystalline Diffraction Beamline for Life and Material Sciences with Modular Microfocus Option 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**

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**Makromol. Chem., Macromol. Symp., 15,79 (1988)*

1. Background

Simultaneous small and wide angle X-ray diffraction (SAXS/WAXS) techniques allow one to investigate at different length scales the structure and dynamics of a wide range of systems of interest in medicine, biology and materials science, 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 the following. During the experiment, two position-sensitive detectors are placed in different locations covering a wide angular range, such as $\sim 100 \mu\text{rad} < \theta < 0.5 \text{ rad}$ where 2θ is the scattering angle. This means that about four orders of magnitude in $q = (4\pi/\lambda)\sin(\theta)$ are accessible. Whilst WAXS provides information about the molecular and atomic ordering of the material, SAXS is sensitive to heterogeneities in the electron density on a larger scale ($1\text{-}10^2 \text{ nm}$). Nowadays, a step forward in this context requires the registration of such information with millisecond time resolution, and with a real spatial resolution down to microns or even nanometers. Beam sizes of a few microns or sub- μm beams can have an enormous impact in fields such as life and materials science, including micro and nanotechnology.

The proposed beamline is expected to be a high resolution and high brightness beam provided by an insertion device at ALBA, generating a high brilliance x-ray beam of the order of $10^{17}\text{-}10^{18}$ photons/s/(mrad)²/(mm)². The beamline should deliver a low divergence highly collimated beam of about $10 \mu\text{m} \times 100 \mu\text{m}$ (vertical and horizontal, respectively) in an energy range optimized around 12.4 keV. A modular microfocus optical system will provide beam sizes in the μm and sub- μm range as required. Small and wide angle cameras with associated linear and area detectors for static and time resolved measurements should be available to record the scattered radiation from samples in a wide range of well-defined environments. Its modular arrangement will allow a choice between ultra small angle scattering for large fibrous structures or microfocus illumination with a simultaneous SAXS/WAXS option for materials studies. At present, there is only one beamline which can provide the microfocus option (ID13 at the ESRF), but this beamline lacks capacity to absorb the demand in the scientific community. As an example, in the report on the meeting of the Soft Condensed Matter & Biological Materials of 24-25 April 2003 at the ESRF, the chairman, Prof. P. Fratzl, reported ID13 and ID02 as being the most demanded beam lines on the committee. For neither of these beamlines could all of the highest-rated proposals be satisfied.

2. The Spanish User Community

In Spain there is a very active and well established community on non-crystalline diffraction with synchrotron light in disciplines such as polymer science, colloid science and biological materials. The research effort on the topic is reflected by the relatively large amount of experiments carried out in the different synchrotron facilities around the world including ESRF, DESY, ELETTRA, DARESURY, BNL etc. The Spanish user community, as inferred by the supporting groups of this proposal, has reached a critical mass and includes a significant amount of permanent scientific staff, but also a significant number of young researchers and PhD students, who will guarantee the future of this field in Spain (see annex I for a precise description of some of the supporting groups). The scientific activity of the community is reflected by a significant, stable and increasing number of scientific publications (see annex II for the last five years) in the topics covered below by the scientific case (Fig.1). This is an important contribution to the scientific literature especially considering that Spain does not yet have a synchrotron facility. Moreover, the potential interest for using synchrotron light for non-crystalline diffraction is attracting an increasing amount of researchers (see annex III). Presumably, this interest will grow significantly if the proposed facility is provided on ALBA. 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.

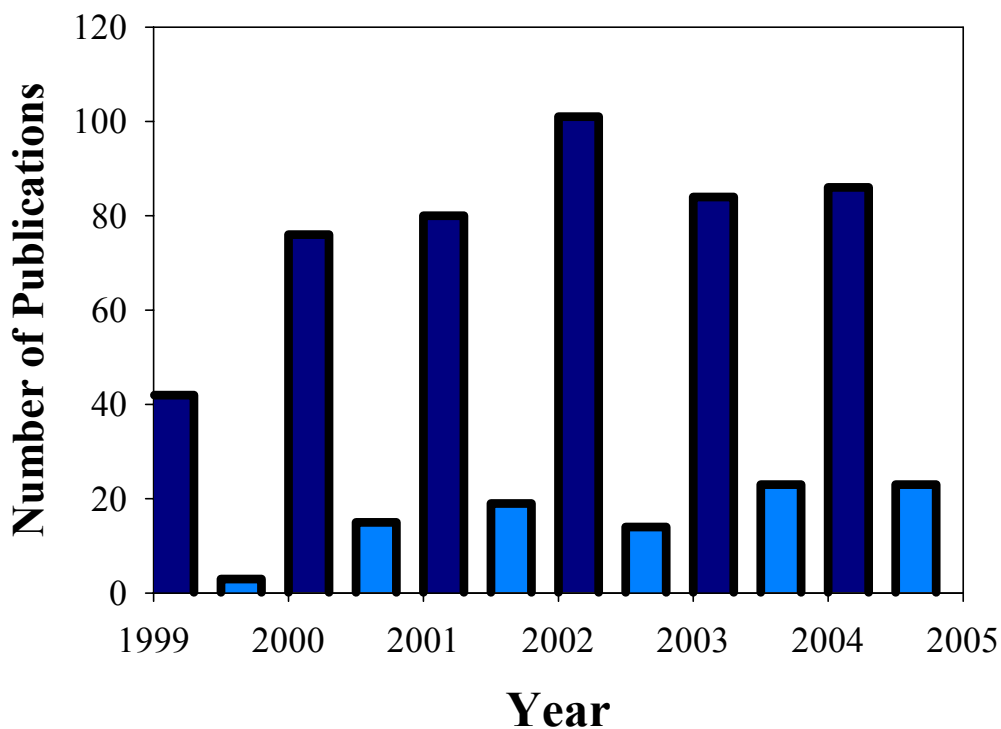


Fig.1: Scientific production of the Spanish User Community in the topics related to the scientific case (dark blue). Those in which synchrotron light was used (lighter blue). The year 2004 is incomplete. The portugese group has also been included.

3. The scientific Case

3.1 Biological systems

Time-resolved X-ray fibre diffraction studies 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 environments 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 comes 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 more than 2300 nm in normal conditions, and muscle tissues are weakly diffracting (fig.2). Consequently, this important biological system can only be successfully investigated with a third generation synchrotron radiation source such as the Spanish source ALBA. 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 allow the measurement of changes in the molecular ordering in space of the muscle proteins with high spatial and time resolution. 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 functions through the conversion of free energy gained from the hydrolysis of ATP.

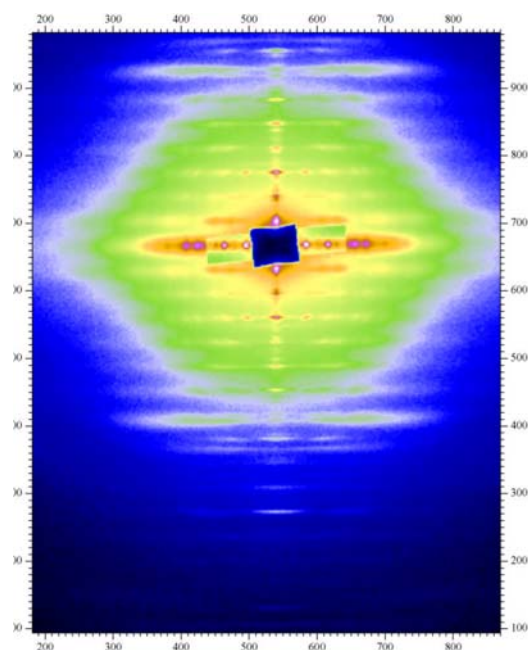


Fig.2: X-ray diffraction pattern from the sartorius muscles of *Rana Esculenta* corresponding to an acquisition time of 200 ms collected at the ID2 beamline, ESRF.

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Time-resolved studies and organization of the photosynthetic apparatus

One of the most used photosynthetic parameters in photosynthesis and plant physiology laboratories is the estimation of the maximum potential photosystem II (PSII) efficiency through measurements of chlorophyll fluorescence. Two different techniques are widely used, modulated chlorophyll fluorescence [1] or continuous chlorophyll fluorescence[1,2] by measuring the shape of the chlorophyll fluorescence induction kinetics during a dark-light transition, the so-called Kautsky effect. During a dark-light transition, the fluorescence level first increases and then decreases with a time scale of seconds and minutes, respectively. Although it is well known that the O to P changes are reflecting mostly the closure of the PSII reaction centers and the P to T changes are dominated by the development of non-photochemical quenching (related to PSII photo-protection), the molecular origin of these changes is not completely known. The use of simultaneous measurement of chlorophyll fluorescence yield (Φ) and lifetime (τ) approached the elucidation of the origins of the different levels of fluorescence during the Kautsky effect [2,3]. Moise et al. (2004) conclude that the differences found between Φ and τ during the Kautsky effect (i.e., the curvature of the $\tau\Phi$ relationship) are due to a variable and transitory non-photochemical quenching [3]. They tentatively proposed that this

quenching results from a conformational change of a PSII core antenna pigment-protein complex during the IP phase. Such conformational changes within the photosynthetic apparatus are susceptible to study by simultaneous small and wide angle X-ray diffraction (SAXS-WAXS).

X-ray crystallography has revealed the structures of some protein complexes in the photosynthetic membranes in remarkable detail, some from photosynthetic bacteria [4] and other from higher plants [5]. Atomic force microscopy allows one to investigate whether X-ray structures are true representations of the proteins in a natural membrane. In the photosynthetic bacteria *Rhodobacter sphaeroides*, a network of linear clusters of specialized chlorophyll proteins is linked together by dedicated light-harvesting proteins that form energy conduits only 20–30 nm wide [6]. As previous authors have stated “it is possible that under certain physiological conditions other organizations may occur”. Light-harvesting complexes, such as the major LHCII from higher plants (see Figure 3 for structure) and other minor complexes, experience conformational changes that play an essential role in the development of the non-photochemical quenching, a process whose molecular basis is not fully understood. A SAXS-WAXS line in the future Synchrotron ALBA would possibly contribute to elucidate the organization of the photosynthetic apparatus of photosynthetic bacteria and higher plants. Also, it would be used to further understand the non-photochemical quenching processes, one of the most important regulation mechanisms in higher plants.



Figure 3. LHCII structure (image obtained from the web; <http://bio.winona.msus.edu/berg/IMAGES/LHCII.gif>).

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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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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 encephalopathies 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 encephalopathies. The degree of primary homology between β -amyloid peptides and prions is very low. However, the similarities of the amyloid structures that both types of proteins can form, has led to 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. Subsequently, it is believed that amyloid fibrilization evolves via a nucleation process, but the structure of the nucleus (or protofibrils) and of fibrils is not

yet known in detail. On the other hand, 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 is of particular interest. 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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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 more detailed information and in which no sample manipulation take place. Some studies on lipid organization have been carried out by X-ray scattering using low energy sources, however the long time exposure of the sample to the X-ray beam makes the technique inappropriate [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 is considered to be crucial for the skin barrier function and its presence is strongly dependent on the lipid composition [5,6]. Recently a controversy has emerged 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 to the presence of microdomains in the lipid structure that could be resolved by simultaneous use of SAXS and WAXS microdiffraction. The microdomain size could be of the order of a few micrometers, therefore a 1 micrometer x 1 micrometer beam size at the sample would be required to resolve this question. The use of a microfocus beam is also necessary to gain correct information on the spatial arrangement in these systems, whether they correspond to a mixture of structures or to separate domains. In fact synchrotron x-ray microdiffraction has resulted very appropriate for the study of similar systems [7], and the reduction of the focus size in the proposed microfocus beam could provide even more interesting results in the field of biological nanostructured systems. Sample stage micropositioning, including tilting, would be required for these studies as well as temperature and humidity control. The detection system should allow for 2D simultaneous recording of SAXS and WAXS.

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3.2 Materials science

3.2.1 Polymer Science

Polymer Crystallization: necessity for millisecond resolution and microfocus

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 order to resolve this problem it is necessary to perform experiments using a third generation synchrotron light source employing detector with a low noise level enabling <10 ms time resolution[8]. In this way, weak molecular ordering events occurring in the induction period of crystallization could be precisely characterized. 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 [9,10]. 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 [11]. The sample can be scanned through the beam along a line with μm -range steps (see the fig. below). A small X-ray beam divergence (less than about 0.2×0.2 mrad²) allows recording both the WAXS and SAXS signals together in the same pattern [12]. This would allow one to perform innovatively accurate data analysis.

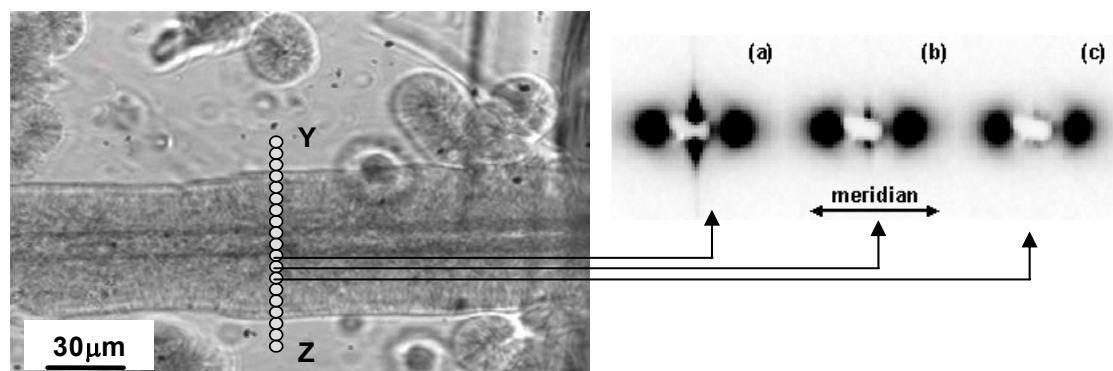


Fig.3. Optical micrograph of a perturbed semicrystalline i-PS sample, showing schematically a linear raster-scan with $5 \mu\text{m}$ raster-increment through the perturbed zone (line YZ).

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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 self-associative phenomena of potential interest in biomolecular recognition [3, 4]. A 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 is 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 that for conducting discotic liquid crystalline polymer filaments the skin presents a higher degree of orientation than the core [6]:

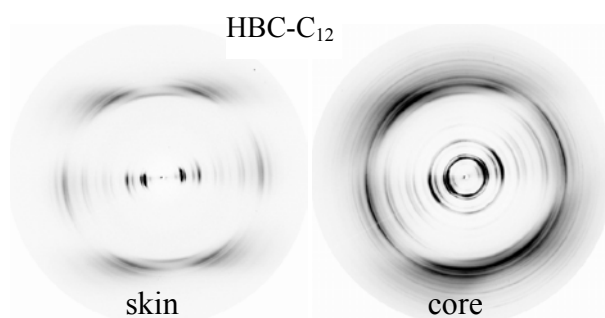


Fig.4. WAXS patterns of a discotic liquid crystalline polymer filaments taken in the skin and the core.

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Multi-component materials: Polymer blends & Microstructure of interphases

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. 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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Multi-component materials: Polymer foams

Closed cell polyolefin foams are two phase materials in which contiguous air bubbles are entrapped in a macromolecular phase (figure 1). These heterogeneous materials have found a wide range of applications. It is widely accepted [1,2,3] that the physical properties of these materials depend on factors such as density, chemical composition, cellular structure and polymer morphology. Nowadays, there are more or less detailed studies about the effect of density, chemical composition and cellular structure [1,2]. However, the effect of the polymer morphology has not been completely understood yet.

In order to use the existent models to predict the physical properties, the properties of the polymer which comprises the cell walls of the foams should be understood. However, these properties are not known, the properties of the same polymer crystallised in a solid sheet are often used instead. The polymer in the foam crystallises in exceptional conditions, being a crosslinked stretched thin film (2-10 μm thickness) crystallised in the presence of a gas. Therefore, it can be expected that the polymer in the cell walls will have a different morphology and consequently different properties to those of a solid sheet of the same material. A previous paper using microdiffraction with synchrotron radiation [4] analysed the morphology and deformation behaviour of a single strut of open cell polyurethane foams showing some interesting and unexpected features of this amorphous polymer. The case of polyolefins the situation is even more complex due to the closed cell structure and semi-crystalline nature of the polymer matrix.

The long-term aim is to characterise the morphology of the polymer in single cell walls and struts of a collection of polyolefin foams with different densities [5,6] and chemical compositions. This morphology would be compared with that of solid sheets of the same materials. The final aim is to understand the relationships between the structure and properties for these foamed materials, a critical aspect in the development of new materials with improved properties. It would be also possible, for the first time, to obtain realistic information on the morphology of semi-crystalline closed cell foams.

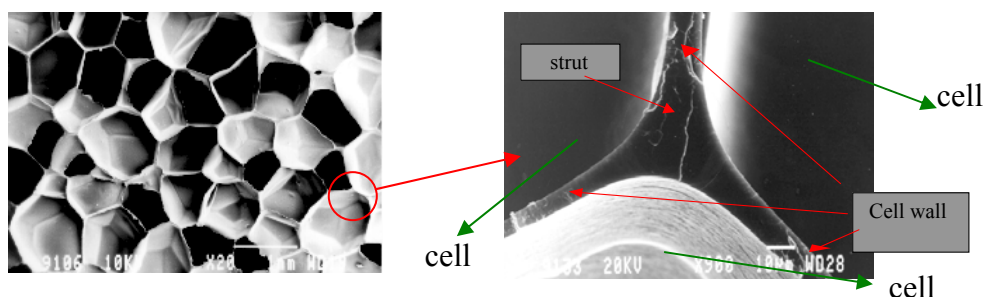


Fig.5. a) General view of the cellular structure of a typical closed cell foam, b) Cell walls and strut

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Multi-component materials: Block and graft polymers

Block copolymers [1,2,3] are of great scientific interest due to their self-assembled supramolecular 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 them as potential candidates as templates in nanotechnology [4]. Besides amorphous-amorphous diblock copolymers, liquid crystalline-amorphous and semicrystalline-amorphous diblock copolymers have obtained substantial attention, because molecular and supramolecular 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, 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]. Moreover, the use of a microfocus facility may enable characterization of individual phases as well as interfacial regions.

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3.2.2 Polymers under external fields

Mechanically induced structure modification

Synchrotron X-ray sources can be used to monitor *in-situ* the deformation processes in polymeric materials [1,2] in order to characterize: i) the evolution of the crystalline phase morphology; ii) craze formation and development; iii) cavitation/voiding phenomena; iv) crack tip dynamics; and v) interphase deformation. In fact, these types of studies require the use of multi-scale length structure sensitive techniques (e.g., simultaneous WAXS and SAXS), high time resolution (mainly for high strain-rate deformations) and high spatial resolution (due to the localized nature of the events). The understanding of the deformation process and the establishment of structure-property relationships are of paramount importance since it allows: i) the optimization of processing methods (e.g., those involving stretching); ii) the maximization of end-user mechanical performance; iii) the development of advanced materials and processes; and iv) designing and customizing materials to specific functions. Experiments carried out using simultaneous SAXS/WAXS using synchrotron radiation in combination with a symmetrical tensile stretching machine allows one to measure the stress-strain curve and the corresponding changes in microstructure in real time during uniaxial deformation. As an example [3,4], the figure below shows the deformation of an initially isotropic block copolymer containing segmented poly(butylene terephthalate) and poly(tetra methylene oxide). The impact of these measurements on material performance and mechanical failure is self-evident.

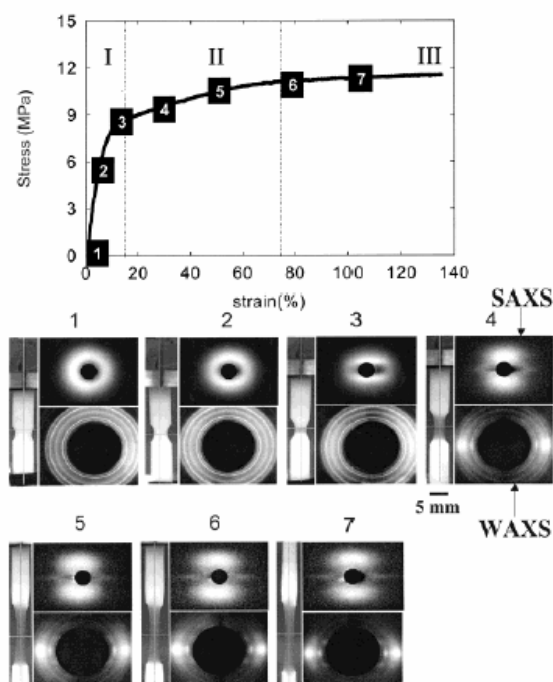


Fig.6 Stress-strain curve for PBT-PTMO copolymer at room temperature. The panels show, for specific values of strain and stress, and the simultaneously collected WAXS (upper) and SAXS (lower) patterns. On the left-hand side of each panel is the corresponding photograph of the sample during drawing.

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Portuguese group contribution:

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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 a sub-100 nm scale need to be developed if increased computing power is 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 μ SAXS and μ WAXS experiments will be of interest in order to spatially resolve the molecular order induced at different length scales. In addition, *in situ* scanning μ SAXS 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.

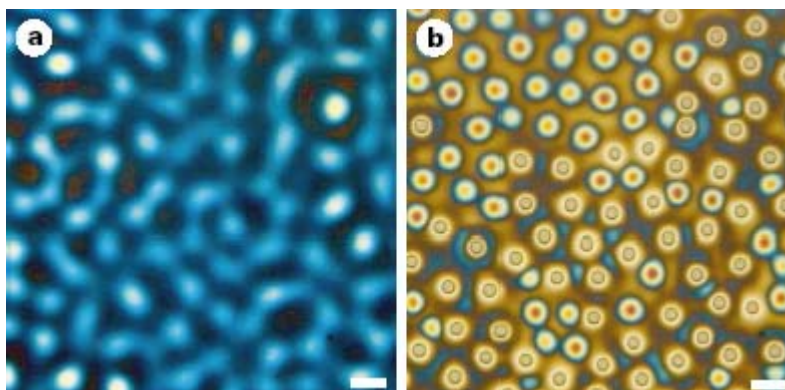


Fig.7. Optical micrographs of polystyrene films which have been exposed to an electric field. In a and b, a 93-nm-thick polystyrene film was annealed for 18 h at 170 °C with an applied voltage $U = 50$ V. The colours arise from the interference of light, and correspond to the local thickness of the polymer 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 the 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].

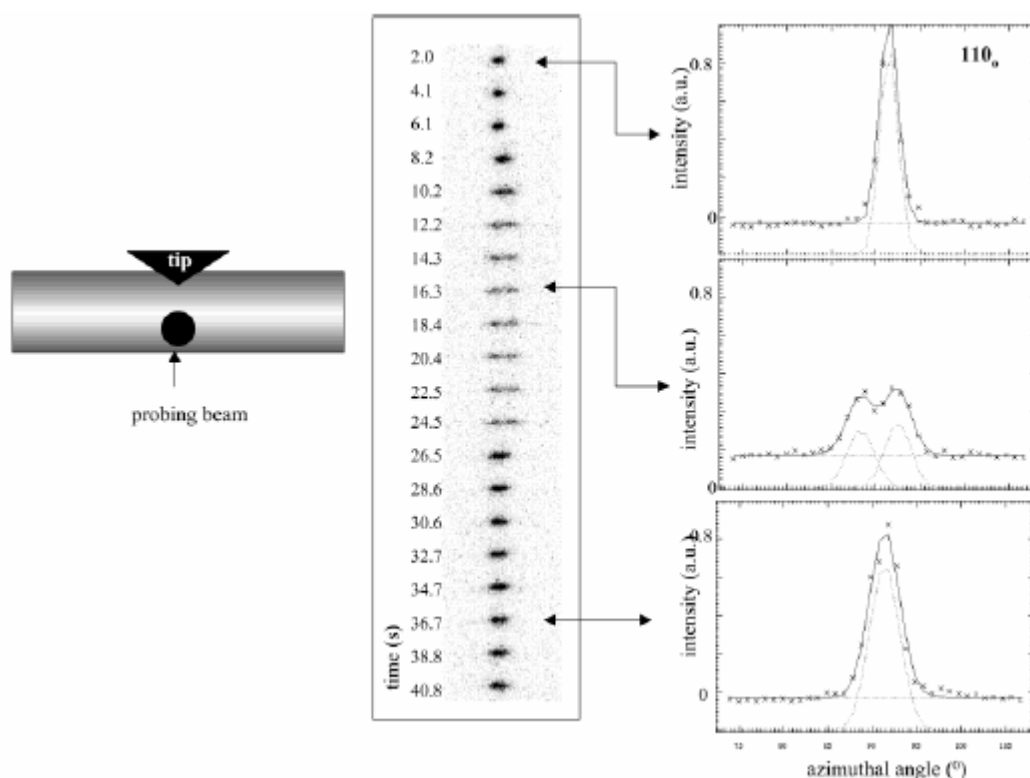


Fig.8 (A) Schematic design of in-situ microindentation experiment on an UHMW-PE fiber. (B) Evolution of 110 orthorhombic reflection during microindentation: at a critical force the 110 reflection splits up into two domains returning to a single domain when the indenter is retracted. Selected azimuthal 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 yet been explored in detail.

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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Non-conventional polymer processing

Polymer structure and morphology are determined, to some extent, by the thermomechanical environment imposed during product manufacture. In fact, the control of process-induced morphology is a convincing route to enhance the mechanical behaviour of polymeric materials[1-2]. This can be achieved by non-conventional injection moulding techniques, where in-mould shear manipulation of the melt during the solidification phase originates a high level of molecular / fiber orientation and constrains the crystalline structure development. SCORIM and PUSH-PULL techniques only differ in the mode of application of the in-mould shearing, resulting in a typical microstructure featuring several highly oriented outer layers and a central spherulitic core[1-2] (Fig.1). The novel RCEM

mould opens new possibilities for the control and manipulation of polymer morphology[3], originating unusual microstructures. The underlying principles governing structure development at all length scales need to be understood in order to enable materials design and customization. X-ray synchrotron sources with microfocus facilities are therefore a fundamental characterization tool, as they allow real time measurements with high real-space resolution.

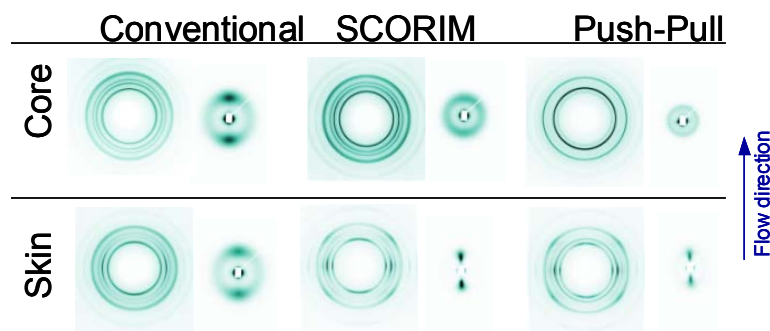


Fig. 9 - WAXS and SAXS patterns of the skin and core layers of injection mouldings: conventional injection moulding, SCORIM (shear controlled orientation in injection moulding) and Push-Pull.

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3.2.3. Microdiffractometry

The advent of synchrotron radiation sources has provided sufficient flux and beam qualities for crystal structure determinations from micrometer-sized crystals (or “microcrystals”) [1]. With the development of third generation synchrotron radiation sources, very intense hard X-ray beams in the μm -range have become available with an acceptable divergence for single crystal experiments [2]. This allows one to probe very small crystals of less than $250 \mu\text{m}^3$ [3,4] and in exceptional cases sub- μm^3 volumes [5]. One can therefore select crystals from powder grains (even from heterogeneous samples) for data collection and structure determination. This is particularly interesting for cases where structure determination from powder data was previously unsuccessful, or where Rietveld analysis does not provide the required structural information. In case of polymer materials, depending on the polymerisation conditions, crystalline microparticles have been observed[6] and investigated at the microfocus beamline ID13 of ESRF [7]. It is expected that single microcrystals of many soluble polymers could be also investigated by microdiffractometry.

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3.2.4. Micro and Nanotechnology.

Nanocomposites in food packaging

Polymers used in the food-packaging sector where outstanding gas barrier properties to oxygen and organic compounds (solvents and food aromas) are required are of special interest. The major drawback of these materials is their moisture sensitivity that causes a significant decrease in their gas barrier properties at high relative humidities [1–3]. Hence, most commercial applications are designed as multilayer structures, where the polymer is sandwiched between highly hydrophobic materials such as polyolefins. Although, these structures provide high barrier properties, the present trend to commercialize extended shelf-life preservative-free food products is promoting a continuous search for enhanced barrier materials. The addition of nanoclays is thought to result in ultrahigh barrier properties mainly due to a tortuosity driven decrease in molecular diffusion of gases and vapours and in increased thermal resistance. The characterization of these kind of nanocomposites using x-ray scattering methods allows one to extract information about the degree of exfoliation and intercalation of the clay platelets which may control properties such as the thermal resistance, glass transition temperature, crystallinity and barrier properties to oxygen [4].

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Carbon nanotube reinforced composites

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 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. 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 μ WAXS and μ SAXS 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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Nanostructured Composites Based on Thermoplastic Polymer Blends

Over the last few decades, short glass- or carbon-fiber-reinforced composites have found widespread application as injection-molded components in the automobile and in other technical industries. The practical importance of these composites is beyond any doubt. However, they are inherently heterogeneous since the matrix is a thermoplastic resin (organic material) in which an inorganic reinforcing phase is embedded. The latter may cause a faster wear of the processing equipment, as well as some problems in recycling whose importance is expected to grow significantly in the near future. Unlike the classical glass- or carbon-fiber-reinforced polymer-based composites, in recently developed nanostructured polymer-polymer composites (NPPC) the reinforcing elements are fibrils of flexible, organic macromolecules embedded in a thermoplastic, isotropic matrix[1,2]. Both the fibrils and the matrix are created *in situ*, through (i) orientation by drawing of a solid blend consisting of polymers with different melting ranges and (ii) selective melting of the major phase component, keeping the temperature below the melting point of the higher-melting component thus preserving its oriented microfibrillar structure. Depending on the chemical functionality of both the matrix and the fibrils, chemical reactions may take place resulting in the formation of a copolymeric interphase. The latter plays the role of a compatibilizer[3]. The mechanical properties of lab-scale NPPC are quite promising[4]. Compression molded NPPC exhibit Young's moduli and tensile strengths of up to 30–50% higher than the weight-average values of the components. The values are comparable to those of short glass-fiber-reinforced composites having the same matrix. This makes NPPCs attractive for many industrial applications. Our goal is to turn the NPPC technology into a cost-effective, large-scale process for transformation of virgin, waste and recycled thermoplastics. This will require a great deal of research in order to establish the optimal relation between the nanostructure of NPPC and their mechanical properties. Our previous measurements[5,6] at the A2 beamline of HASYLAB and ID13 of ESRF have shown that simultaneous and microfocus SAXS/WAXS measurements are fundamental for the optimization of the stage of selective matrix isotropization of NPPC, for choosing an adequate injection molding technique, as well as for determining of its conditions.

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Nanofilms: Ordering phenomena in confined environments

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 applications in microelectronic and 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.10 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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Spanish group contribution: ref. 8

3.2.5 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 μm), 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, the adsorption-desorption rates being very rapid.

An issue of special relevance for the characterization of the ACF is their fiber shape, since it introduces differences in 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, a result of the activation process (i.e., activation temperature, activation method, activating agent) and the precursor used. Although a remarkable research effort has been made 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) offers some advantages for the characterization of the porosity in activated carbons, since it is sensitive to both closed and open porosity. Another important advantage is that SAXS intensity profiles are sensitive to the shape and orientation of the scattering objects, so 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 micrometer size beams, via the micro-SAXS technique. The high intensity of the beams that can be obtained nowadays should also allow studies with a sufficiently high temporal resolution, 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, which is the relationship between porous texture and properties of the ACF, such as adsorption and mechanical properties, as previously mentioned.

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 [1-4]. These studies have been very useful to understand the differences among preparation methods and precursors. There is still important work to be done in this area, especially regarding the use of this technique to follow the development of porosity during the preparation process, in-situ and with a spatial resolution close to micrometer.

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3.3 Colloidal systems

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 on 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 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 either by 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 some insight on the conditions in which these controlled emulsification processes are successful, or in which they fail.

In this respect, the construction of a beam line specialised in micro focus scattering-diffraction would be useful to perform experiments in which two liquids are in contact. This would allow one to follow the kinetics of structural transitions in real-time and 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 the mean after mixing can be achieved [6, 7]. Also experiments in which a solid is in contact with a liquid could give information on the dynamics of solubilisation of materials deposited on the substrate. The need for the microfocus originates in both the distance resolution of the experiment and the possible inhomogeneities that can be established in the systems. Thus, a beam with size of 1 micrometer, at least in one axis, would be required. In order to measure kinetics the beam intensity and detector time resolution should be adequate for a time resolution of the order of 10^{-4} seconds in a 2D SAXS detector. Sample positioning should be resolveable to better than 1 micrometer. These experiments would require the use of both stopped flow and continuous flow equipment.

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Microfocus approximation to single particle scattering/diffraction

The use of a microfocus 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, it is not always possible to achieve a high dilution without changing the structure of the system. The use of microfocus can allow the study of the form factor of single particles. The investigation of single particles also has the advantage that structures can be resolved without the size polydispersity that always obscures the form factor information. If the time resolution is not adequate, immobilisation techniques, either on a solid substrate or by increasing the medium viscosity, would be needed. The systems where this technique would be applicable correspond to any area of colloid science. In particular, information about the mechanism of liposome or emulsion solubilisation could be improved [2], and 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, however, reaching sub-micrometer beam size would be particularly useful for single particle scattering-diffraction. A sample of 0.1 mm thickness containing 5mg/L of particles with 100 nm radii would require a beam of 1micrometer x 1 micrometer. The detector should allow for acquisition times below 0.1s and the flux at the sample should be enough for detection of single particle with reasonable signal.

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Phase transitions induced by temperature jumps in colloidal systems.

A temperature jump, during heating or cooling, can induce important changes in the structures present in surfactant based colloidal systems. The characteristics of the jump and of the phases crossed during the jump can lead to important differences in the final (non-equilibrium) state of the system. This is fundamental, for instance, in the formation of emulsions by temperature change. Depending on the speed of heating, the droplet size of the emulsion formed can be very different or, the emulsion cannot form, reaching instead a different aggregation state. In the particular case of highly concentrated water-in-oil emulsions, we have observed the formation of intermediate states in the system by time resolved X-ray Scattering measurements performed at ELLETRA. The time evolution of the system during a temperature jump is shown in the figure. Although with the time resolution achieved of 0.5s, hints on the correspondence of the non equilibrium structures with the equilibrium structures could be made, detailed differences between these two different dynamic and static structures could not be observed. The final structure of the emulsions depends on the details of the jump speed, but no detailed conclusions could be drawn at this point.

Other systems where a temperature jump can play a role is on temperature induced vesicle formation, and in general on the formation of any non-equilibrium structures. The time resolution of the experiment should be enough to assure the detection of intermediate structures with adequate statistics. Thus, high dynamic range detectors (about 5 orders of magnitude) with time resolution of about 1ms should be available to improve these experiments.

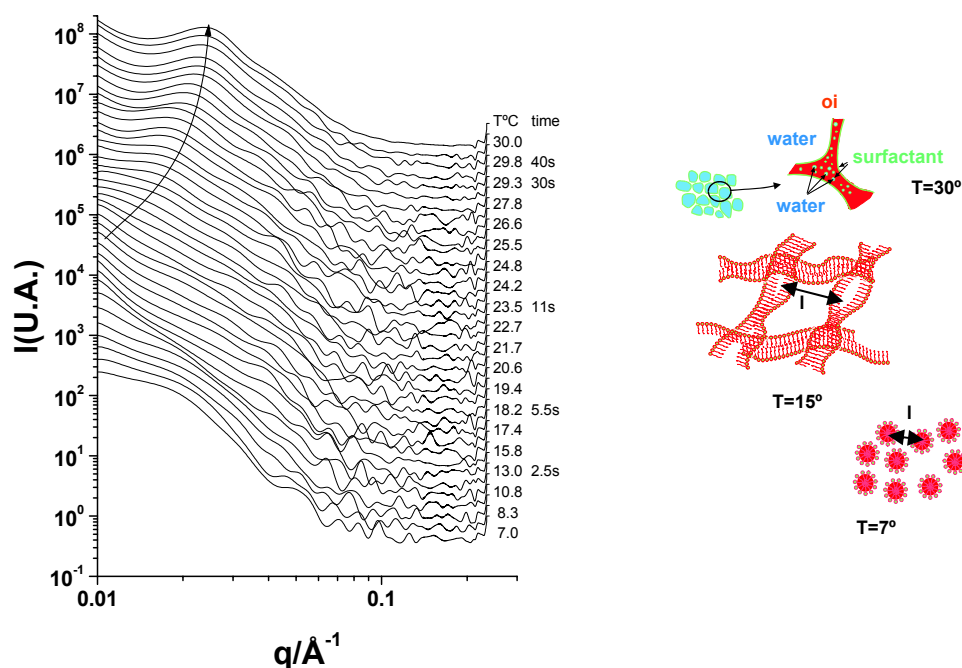


Fig.11. Evolution of the x-ray scattering curves for the composition 92% H₂O, 3% C12EO9 and 5% C10H22. The line is a guide for the movement of the correlation peak. At low temperature the scattering curve is compatible with O/W microemulsion ($R=12.0\text{nm}$), at intermediate temperatures a bicontinuous structure is compatible with the curves (lamellae 7.5nm thickness) and at high temperature a highly concentrated water-in-oil structure is compatible with the curves. The correlation peak corresponds to the continuous phase structure and the Porod behavior at low q corresponds to the emulsion droplets scattering ($R>100\text{nm}$) [1,2].

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Dynamics of phase transitions induced by compositional change

This kind of process is widespread in technological applications; from paint drying to the emulsification of concentrates or to vesicle reconstitution by dilution. The dynamics and phase changes during the process will influence the micro and nanostructure of the final composition if this corresponds to a metastable non-equilibrium state [1]. Knowledge of non-stationary stages will allow the control of the final properties. This is the case of miniemulsions formed by microemulsion dilution with excess water. Preliminary experiments in an in-house Kratky SAXS camera coupled with a home-built continuous-flow stopped-flow mixing device are shown in the figure [2].

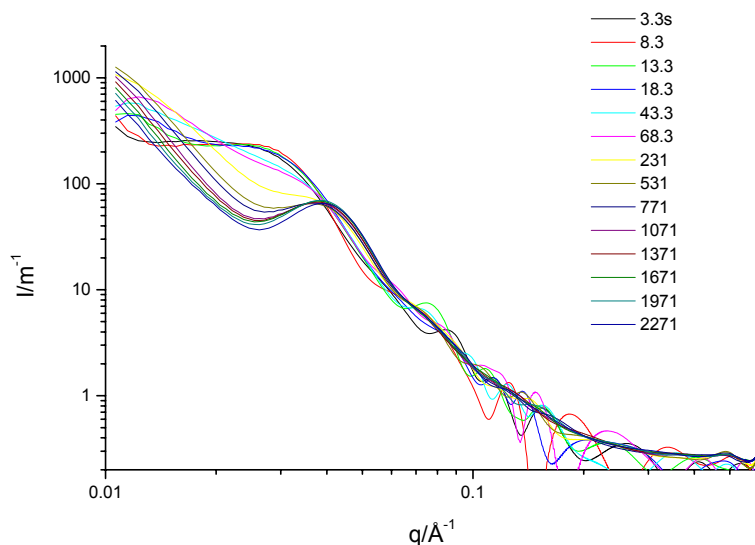


Fig.12. Time resolved experiment in a microemulsion.

In the case shown in the figure, a time resolution of about 5 s seems enough to study the system. Systems like fast coagulation of colloidal particles would need shorter time resolution, probably below 0.1 second.

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Role of the electrostatic charge on the kinetics of the first steps of liposome-surfactant interaction: adsorption of surfactant and desorption of mixed micelles

The solubilization of liposomes by surfactants has been described as a three-stage process: vesicle saturation, formation of mixed micelles and complete solubilization [1,2]. However, the initial fast steps of solubilization, which consist of the adsorption of surfactant on the membrane and desorption of mixed micelles from the membrane, are not still clear due to the lack of techniques with sufficiently short experimental timescales. The study of the effect of ionic lipids on the kinetic of these processes is essential for two reasons: biological membranes contain ionic lipids and a number of biological processes are related with mechanisms of adsorption (fusion, endocytosis, viral infection, etc) and desorption (exocytosis of synaptic vesicles, secretion of membranous vesicles, etc) [3].

Experiments performed at the ELETTRA Synchrotron in Trieste point out that if surfactants and lipids have the same type of electrostatic charge the adsorption of surfactant on the liposome is slower and the release of mixed micelles from the liposome surface is faster than when the species are oppositely charged [4,5]. Thus, it seems that the electrostatic charges could either accelerate or slow down these processes. However, in order to confirm this hypothesis future research should consider experiments in which the amount of charge, the lipid composition and the type of surfactant mimic the real biological membranes conditions. In this respect, the small angle x-ray scattering (SAXS) technique for time resolved experiments using a stopped flow cell and synchrotron radiation is required. The beam intensity and detector time resolution should be adequate to achieve a time resolution of about 10^{-4} s and stopped flow and / or continuous flow devices are also required. The use of this sensitive methodology opens up new possibilities for the control of processes containing surfactants and lipids from both biological and physical-chemical perspectives.

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4. Requirements

At present there are several beamlines in the world involved in non-crystalline diffraction, for example, 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 a microfocus facility.

The nature of the user community, with a broad spectrum of research interests ranging from biological to polymer materials, strongly suggest the need for a beamline at ALBA dedicated to non-crystalline diffraction with high brightness, high spatial and time resolution, providing in addition a microfocus facility. The incoming radiation should be provided by an insertion device located in an adequate segment of the machine in order to cover the characteristics of the beam, presented below. Microfocus should be provided as a module to be inserted when either illumination of very small samples or high spatial resolution are required.

Characteristics of the beam

Insertion Device: According to the initial calculation on the lattice for ALBA (<http://www.cells.es/home2.html>) five different types of insertion devices have been considered including two types of wigglers and three types of undulators. 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.

Brilliance: 10^{17} - 10^{18} photons/s/(mrad)²/(mm)²

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}

With standard focusing

Beam size at sample/detector: 10 μ m (V), 150 μ m (H)

Divergence at sample/detector: 0.02 mrad (V), 0.04 mrad (H)

With microfocus

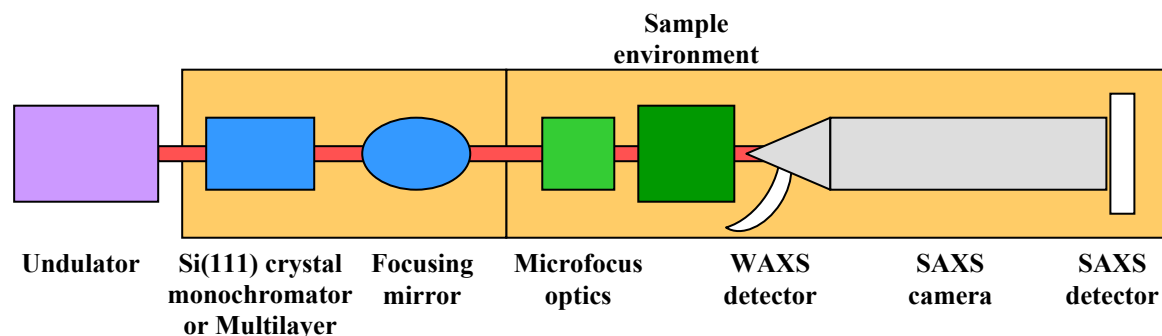
Beam size at sample/detector < 5 μ m diameter.

Divergence at sample/detector: $\approx$ 0.05 mrad

Positional stability: 1 % RMS during 1-10 seconds

4. The tentative layout

A tentative scheme of the beamline has been outlined below:



Undulator

- In vacuum undulator. The characteristics of the undulator will be determined at CELLS.

Optics

- A double crystal Si (111) monochromator.
The possibility to have available and to employ optionally two monochromating systems either the classical based on Si(111) or a multilayer system in order to select the bandpass must be considered during the design phase.
- A toroidal focusing mirror (vertical and horizontal focusing).
It should be noted, that X-ray optical systems for SAXS/WAXS applications are in rapid evolution at ESRF and the current scheme of focusing mirror optics might be superseded by other optical systems such as refractive optics. Such an optical system would allow keeping the beam on-axis with the possibility of a beam-path deviation by flat double-mirrors for background reduction.

Microfocus

- Pin-hole collimation (5 and 10 μm collimators).
- Kirkpatrick-Baez mirror. Commissioning studies on ID13 indicate that a KB mirror can provide a beam $\leq 1 \mu\text{m}$ (depending on position) with a flux of 10^{11} ph/s.
- Fresnel lenses (0.1-0.5 μm with a 10^9 ph/s flux).
- Beryllium Compound Refractive Lenses. Focusing properties depend on the number of individual lenses and spot sizes of a few microns can be easily reached at the current stage. This would allow optimising either for spot size or divergence.

Sample environment

At this moment in time we foresee the need for the following features but the list may well be extended or be modified in due course.

- Scanning set-up: - x/y translation stage: load capacity 50 kg, travel range 100 mm.
- Micro-hexapod: rotation range $\pm 5^\circ$ with 2 μrad increment, travel range ± 6 mm with 0.1 μm increment (x/y/z), load 2 kg.
- Video microscope.
- Microgoniometer: rotating arm; 5, 10, 30 μm beam; horizontal sample ϕ -axis; on-axis sample observation.
- Temperature furnaces.
- High pressure cells.
- Heating/cooling stage for DSC (Linkam, Mettler, etc)
- Heating/cooling shear cell.
- Tensile stretching machine.
- Magnetic field.
- Electric field.
- Stopped flow and continuous flow equipment.
- Others

The Detectors

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

- A fast single photon counting area detector, area approximately $20 \times 20 \text{ cm}^2$, covering a large (infinite) dynamic range with a maximum count rate of 10×10^6 counts/sec, spatial resolution of 200 – 250 μm and an efficiency between 60 – 80 % depending upon the actual wavelength chosen is *required*. Note: Professor Yagi at SPRING 8, Japan, has for his high intensity beam line just bought a detector with these specifications, the RAPID detector from Daresbury Laboratory (UK), as it is the only today available device that meet these specifications and is suitable for time resolved studies in e.g. life sciences where processes of interest occur on a sub-millisecond time scale.

- CCD Area detector for SAXS and WAXS (MAR CCD, diameter ~ 300 mm)
- Fast linear one dimensional detectors ($\approx 4 \times 10^5$ counts/s/channel): for WAXS (1-D microstrip gas chamber curved arrangement) and SAXS (1-D wire detectors).

The Experimental Hutch

We foresee that we will need a hutch that has a minimum height of >2.5 m or more. This in order to allow the SAXS/WAXS camera of a minimum length of 10 m to be oriented horizontally as well as pivoted up/sideways to a certain degree. We must also allow for a crane, chains, runners and its hook to move freely above the experimental configuration consisting of sample stage, camera, detector and extra instrumentation used by our user group and hinted at under Sample Environment.

We need a hutch that is ≥ 3 m wide or wider at the junction between the optical and experimental hutch whilst assuming that only the gird slit and sample stage (including added instrumentation identified under sample environment) is located in the hutch. We want to see this width increasing the further away from the source point we go in order to take advantage of the spreading out of the fan.

We finally need a hutch that is greater than > 12 m in length. In this area we expect to mount a SAXS camera with a radius of 0.5 m and a minimum length of 10 m. In addition, we must fit in the sample stage immediately inside the experimental hutch wall separating experimental and optical hutches and we must ensure that we have at least one meter of space at the end of the camera available to mount detectors, bring in equipment into the hutch that may be 'bulky', to allow for manipulation of the crane and the possible collection of equipment at the rear end that may need to be transported towards the front etc by the crane.

ANEXO I: Precise description of some of the supporting Research groups.

- 1. Dynamics and Structure of Soft Condensed Matter Group. Instituto de Estructura de la Materia, CSIC. Serrano 119, 28006-Madrid**
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ANEXO II: Selected publications of the last five years related to the scientific case.

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1. I. Campoy, M.A. Gómez, C. Marco. Isothermal crystallization of nylon 6/liquid crystal copolyester blends. *Polymer* 40, 4259, 1999.
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ANEXO III: Potential users.

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2. Prof. C. Vázquez Vázquez. **Universidade de Santiago de Compostela. Facultade de Química. Dpto de Química Física. Avenida das Ciencias, s/n, E-15782 Santiago de Compostela .** qfmatcvv@usc.es
3. **Departamento de Bioquímica y Biología Molecular. Facultad de Ciencias. Universidad Autónoma de Barcelona.**
 - Prof. P. Suau, University full Professor
 - Dr. I. Ponte. University Profesor
4. Prof. F. Monroy Muñoz. **Dpto. Química Física I. Facultad de Química. Universidad Complutense de Madrid.** monroy@quim.ucm.es
5. **Universidad de Granada. Dep. of Inorganic Chemistry**
 - Isidora Bautista Toledo
 - Francisco Carrasco Marín
 - David Fairén Jiménez
 - Mari Angeles Ferro García
 - Francisco J. Maldonado Hódar
 - Carlos Moreno Castilla
 - Agustín F. Pérez Cadenas
 - Jose Rivera Utrilla
 - Manuel Sánchez Polo
 - Ruth Ubago Pérez
6. **Universidad de Jaén. Dep.. of Inorganic Chemistry**
 - Miguel A. Álvarez Merino
 - Maria Victoria López Ramón
7. M. Prieto. **Centro de Química-Física Molecular, Complexo Interdisciplinar, IST .Av. Rovisco Pais, P-1049-001 Lisboa, Portugal.** prieto@alfa.ist.utl.pt
8. Felix M. Goñi .**Centro Mixto "Unidad de Biofisica", Universidad del Pais Vasco y CSIC. Bilbao.** gbpgourf@lg.ehu.es
9. Joan Estelrich. **Departamento de Fisicoquímica, Facultad de Farmacia, Universidad de Barcelona, Av. Joan XXIII s/n, 08028 Barcelona.** joanestelrich@ub.edu
10. Carmen López-Iglesias. **Servicios Científico-Técnicos, Universidad de Barcelona, Parque Científico de Barcelona, C/Baldiri Reixac 10-12, 08028 Barcelona.** carmenli@giga.sct.ub.es
11. Jesús Guinea. **Departamento de Microbiología, Facultad de Farmacia, Universidad de Barcelona, Av. Joan XXIII s/n, 08028 Barcelona.**
12. Iñigo Angulo Barturen, **GlaxoSmithKline Investigación y Desarrollo, SL, Diseases of The Developing World, Drug Discovery Biology, Therapeutic Efficacy, C/ Severo Ochoa, 2, 28760 Tres Cantos. Madrid.** iab53075@gsk.com
13. Ms. Steffi Bergmann, **NanoBioMatters S.L.CEEI, Parque tecnologico, Av. Benjamin Franklin, 12, 46980 Paterna** sbergmann@nanobiomatters.com
14. José Ignacio Velasco, **Centro Catalán del Plástico. Colom, 114, edificio Vapor Universitari. 08222 Terrassa.** jose.ignacio.velasco@upc.es
15. Manuel Laso. **Departamento de Ingenieria Quimica de la Escuela Tecnica Superior de Ingenieros Industriales, Universidad Politecnica de Madrid.** laso@diquima.upm.es

16. Pedro Poza. **Departamento de Tecnología Química, Ambiental y de los Materiales, Escuela Superior de Ciencias Experimentales y Tecnología, Universidad Rey Juan Carlos. Madrid.** papoza@escet.urjc.es
17. Luis Barral Losada (labpolim@udc.es), María José Abad López Maria Jose Abad (mjabad@cdf.udc.es) **Grupo de Polímeros. Departamento de Física Universidad de A Coruña. Escuela Universitaria Politécnica, Avda 19 de Febrero, s/n, 15405 Ferrol (A Coruña).**

ANEXO IV: Support Letters

- Letter from the Spanish Company REPSOL.
- Letter from the President of the Spanish Polymer Group (GEP) of the Spanish Royal Society of Chemistry (RSEQ) and Physics (RSEF).
<http://www.ucm.es/info/rsequim/gep/>
- Letter from the President of the Specialized Group of Colloids and Interfaces (GECI) of the RSEQ and RSEF. <http://www.ucm.es/info/geci/>
- Letter from the Director of the Institute of Science and Technology of Polymers, CSIC. <http://www.ictp.csic.es>

REPSOL S.A. has shown to us its support to the present beamline proposal. The supporting letter was not received the date of submission of the proposal to the SAC of ALBA (22nd of December 2004). As soon as the letter arrives it will be submitted to the SAC.



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DE INVESTIGACIONES
CIENTÍFICAS

INSTITUTO DE CIENCIA Y
TECNOLOGÍA DE POLÍMEROS

Madrid, November 24, 2004

To whom it may concern

Dear Sir/Madam,

As President of the Specialized Group of Polymers (GEP) of the Spanish Royal Society of Chemistry and the Spanish Royal Society of Physics, I am writing to you to express the support of the GEP to the creation of a beamline in the Spanish Synchrotron to be built in Barcelona.

This beamline should be dedicated to non-crystalline diffraction (SAXS and WAXS facilities included) for the characterisation of soft condensed matter including polymeric materials. This facility is certainly very useful and necessary for the scientific activities carried out by many of the research groups in the GEP and is eagerly awaited by most of the members of the Spanish Polymer Group, already involved in that type of research.

If you need any complementary information about this letter of support, please do not hesitate to contact me at the E-mail: perena@ictp.csic.es.

Sincerely,

José M. Pereña
Research Professor
President of the Spanish Polymer Group (GEP)
Internet: www.gpolimeros.org



Universidade de Vigo

Dr. Luis M. Liz Marzán
Depto. de Química Física
Facultade de Ciencias
36200 VIGO
Tfno: 986 812298
Fax: 986 812556
email: lmarzan@uvigo.es

Vigo, November 23, 2004

To whom it may concern:

Dear Sir/Madam,

In the name of the President of the Specialized Group of Colloids and Interfaces (GECI) of the Spanish Royal Society of Chemistry and Spanish Royal Society of Physics, I am writing to you to express the support of the GECI toward the creation of SAXS and WAXS lines for soft matter characterization, within the Spanish Synchrotron to be built in Barcelona. Such lines would be extremely beneficial for the research being carried out by most of the groups involved in the GECI.

Luis Manuel Liz Marzán
Secretary of the GECI



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November 26, 2004

TO WHOM IT MAY CONCERN

As Director of the Institute of Polymer Science and Technology (ICTP), CSIC, I would like to outline the support of the Institute to establish a beamline in the Spanish Synchrotron facility to be built in Barcelona.

This beamline could be dedicated to non-crystalline diffraction (including SAXS and WAXS facilities) for the characterization of soft condensed matter, i.e., polymeric materials. This facility would be certainly necessary and very useful for the scientific activities carried out by many of the Departments in the ICTP and would eagerly be awaited by most members of the Institute, already involved in that type of research.

If you need any complementary information, please do not hesitate to contact me at the address indicated below.



Sincerely yours,

Leoncio Garrido
Director ICTP

C/ Juan de la Cierva, 3
28006 Madrid ESPAÑA
Telf. 91 562 29 00
Fax.: 91 564 48 53