of the PSCM is to streamline neutron and synchrotron based Soft Matter Research to address 21st century challenges in nanomaterials, biotechnology, environmental and energy sciences. New laboratories equipped for sample preparation and characterization will be hosted in the new Science Building to be soon implemented on-site. This structure should permit to bridge the gap between the ESRF and ILL facilities and the European industry in many issues which require SAS measurements.

Keywords: SAXS, SANS, industry

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SAXS in support of industrial research at DuPont

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DuPont offers a wide range of innovative products and services for markets including agriculture, nutrition, electronics, communications, safety and protection, home and construction, transportation and apparel. The SAXS group within the Corporate Center for Analytical Sciences provides support to R&D and businesses throughout DuPont, now in approximately 90 countries. The SAXS technique is unique for the characterization of particle size and agglomeration/flocculation at the nanoscale, providing statistically meaningful information; this technique is particularly powerful when coupled to a synchrotron. Two examples are provided.

The realization of the full potential for polymeric nanocomposites to manifest their entitled property improvements relies, for some properties, on the ability to achieve maximum particle-matrix interfacial area. Well-dispersed nanocomposites incorporating colloidal silica as the filler can be realized in both polystyrene and poly(methylmethacrylate) matrices by exploiting the charge stabilized nature of silica in nonaqueous solvents which act as Lewis bases. We demonstrate that dispersions of colloidal silica in dimethylformamide are charge stabilized, regardless of organosilyl surface functionalization. When formulated with polymer solutions, the charge stabilized structure is maintained during drying until the charged double layer collapses. Although particles are free to diffuse and cluster after this neutralization, increased matrix viscosity retards the kinetics. We demonstrate how high molecular weight polymers freeze the structure of the silica to produce well-dispersed composites. The glass transition temperatures of these composites do not vary, even at loadings up to 50 v%.

An in-situ Study of the pyrolytic synthesis of titania nano-particles was used to understand the particle growth dynamics in a flame reactor. Particle size (SAXS), chemical speciation (XAS), crystal phase identification (WAXS) were measured as a function of height above the burner to follow the chemical conversion from $TiCl_4$ to TiO_2 and associated nanoparticle growth and agglomeration dynamics. Much of the observed behavior could be tied to separate hydrolysis and condensation reactions occurring in different lateral and horizontal regions of the flame governed by water concentration and temperature. Crystallization kinetics paralleled the kinetics of particle growth indicating that in some cases single grain particles may form directly from the vapor phase. Due to the lateral distribution of water vapor in the flame and the lateral temperature gradient, hydrolysis and titania conversion had a maximum away from the flame axis, while condensation and nucleation occurred primarily at axial positions. This study demonstrated that synchrotron SAXS can be used to study pyrolitic synthesis reactions of industrial interest.

Keywords: industrial, synchrotron, SAXS

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SAXS and SANS for industrial materials-by-design

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Over several decades, absolute-calibrated small-angle X-ray and neutron scattering (SAXS and SANS) studies of material microstructures, measured over extended length scales, have helped overcome technological barriers impeding advanced materials development for industrial applications. While much work has focused on applications of soft materials (new polymers, biological systems, etc.), SAXS and SANS also make significant contributions in the development of new high-performance "hard" materials of industrial relevance:

Advanced thermal barrier coating (TBC) development has allowed more efficient gas turbine performance to be achieved for jet propulsion and electricity generation. However, the thermomechanical properties of these coating materials depend critically on their highly anisotropic component void morphologies. SAXS and SANS studies have succeeded in distinguishing and quantifying the different components, and have elucidated many of the processing–structure–property relationships for improved materials design [1].

Solid oxide fuel cells (SOFCs) promise major improvements in energy efficiency, clean energy supply and fuel conversion. The small X-ray beams available for SAXS and ultra-small-angle X-ray scattering (USAXS) measurements at 3rd generation synchrotron sources, together with the X-ray energy tunability, enable the microstructure gradients and the state of the electrochemical interfaces close to the electrode–electrolyte boundaries to be interrogated. Anomalous USAXS studies have quantified degradation during SOFC service life such as arises from the presence of sulfur in the fuel [2].

Despite more than a century of research, fundamental aspects of cement hydration, particularly the nature of the nanoscale calcium-silicate-hydrate (CSH) reaction product, remain elusive. For the cement industry, this situation presents a challenge to the development of new cements with advanced curing properties, or for achieving reduced CO2 emissions in cement manufacture. Carefully calibrated SANS contrast variation measurements have determined the composition and density of the CSH solid phase from first principles. The atomic packing density within CSH is higher than in related mineral analogs – with repercussions both for cement shrinkage and for new cement design [3].

The global coal industry seeks new sorbent materials to select and remove CO2 from the flue gas streams of coal-fired electrical power plants. Various classes of metal organic frameworks and similar materials exhibit promising sorption properties, but the structure–dynamics–property relationships are complex. SAXS and SANS studies provide a key role in correlating the structural changes measured by small-angle-diffraction both with the observed sorption performance and with the powder morphology or particle surface area [4].

Using these examples the potential of future SAXS and SANS development will be highlighted in the context of new industrial materials design.

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