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THE DEVELOPMENT OF HIGH ENERGY POWDER DIFFRACTION AT SRI-CAT AND THE FUTURE DEDICATED HIGH ENERGY X-RAY SCATTERING FACILITY HEX-CAT AT THE ADVANCED PHOTON SOURCE

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Third-generation synchrotron radiation sources are capable of producing extremely intense beams of high energy x-rays with energies above 35 keV. This has provided a wide variety of important opportunities in diverse fields. Specialized undulators and optics can achieve unprecedented brilliance and photon flux in this energy range.

The use of high x-ray energies for powder diffraction largely eliminates absorption and extinction effects, and opens the possibility of obtaining very high-quality data for materials containing high-Z elements. The ability to penetrate bulk samples or environmental cells enables dynamic studies under extreme and carefully controlled conditions. The short wavelength also allows the measurement of very high Q data, providing more structural detail. High energy x-rays from third-generation sources display many of the advantages of neutrons from pulsed sources, but with much higher brightness and better resolution. We will present the optics and instruments that have been developed for high-energy x-ray diffraction at sector 1 of the SRI-CAT and for future use at the High Energy X-ray Scattering Collaborative Access Team (HEX-CAT) beamlines. Our studies of disorder in clathrate thermoelectric employing high-energy powder diffraction will be used to illustrate both the potential of high energy x-rays and the challenges associated with their use for powder diffraction.

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Keywords: HIGH ENERGY X-RAYS POWDER DIFFRACTION SYNCHROTRON RADIATION

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HIGH ENERGY X-RAYS FOR THE STUDY OF ORIENTATIONAL ORDER IN NONCRYSTALLINE MATERIALS

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Noncrystalline materials are characterized by a loss of translational and orientational order. Conventional scattering experiments measuring the structure factor $S_{(q)}$ allow determination of most of the degree of translational order in noncrystalline materials. Direct information about the orientational order is lost due to averaging in space and time, in particular for liquids. Thus, the local point symmetry of bulk short-range order in simple monatomic liquids has remained one of the fundamental open questions in the science of condensed matter. Using high energy x-rays (E=71.5 KeV) we have recently probed the local symmetry of liquid lead at a heterogeneous solid-liquid interface. The experiments reveal fivefold local point symmetry in the liquid adjacent to a solid Si(100) wall. This unique information has become accessible by exploiting two experimental devices: We capture and align the local liquid building blocks at a non-wet Si(001) wall and observe the point symmetry of the interfacial liquid scattering using evanescent x-ray waves produced by total reflection at the deeply buried internal wall-liquid interface.

Keywords: ORIENTATIONAL ORDER, LIQUIDS, HIGH ENERGY-DIFFRACTION

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SOLID FORM CHARACTERISATION IN THE PHARMA INDUSTRY USING HIGH THROUGHPUT AUTOMATED SCREENING, MANUAL INVESTIGATION AND PROCESS

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During the past five years automation has played an ever-increasing role in the preparation and characterization of solid form in the pharmaceutical industry. Parallel screening tools have enabled the screening of a large number of parameter matrices utilizing current theoretical guidelines in vastly reduced timescales compared to more traditional techniques. This has been matched by advances in high throughput analytical tools and the incorporation of informatics to offer the material scientist improved confidence in screening techniques, productivity and the quality of results, placing scientists in an unprecedented position to make informed decisions. The presentation will discuss the enabling tools that are currently in use at Pfizer, Sandwich and how these are balanced with high quality manual investigations in the process of developing a drug candidate. An explanation of how the automation of processes as well as analysis has prevented the generation of “bottle necks” in a procedure will be provided and how advances in pattern comparison software and informatics have helped produce trends from a large number of experiments.

Keywords: HIGH THROUGHPUT SCREENING AUTOMATION CHARACTERISATION

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HIGH-THROUGHPUT X-RAY DIFFRACTION FOR POLYMORPH SCREENING

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Polymorphism is of increasing importance in the formulation of pharmaceutical products and the protection of intellectual property rights. In other areas of chemical industries, like pigment research and manufacturing controlling polymorphism is essential. Beside these application aspects, polymorphism is also at the center of understanding crystallization and illustrates the subtleties influencing crystal packing. Controlling polymorphism requires knowledge about zones of stability in the phase diagram. This information can be obtained by carrying out a multitude of crystallizations and identifying the resulting polymorph or mixture of polymorphs. A method has been devised which allows for parallel crystallization under varying conditions, followed by automated X-ray powder diffraction without the need for individual sample preparation. The method is demonstrated on the example of anthranilic acid and other organic compounds. Systematic studies varying solvents and mixtures of solvents as well as changing the temperature in the range of 0 to 50°C have reproduced the known polymorphs and established stability trends.

Keywords: HIGH THROUGHPUT SCREENING POLYMORPHISM