# Chapter 1 - **INTRODUCTION**

Nanometer scale structures include sizes from the near atomic (nanometer) scale to the near optical (micrometer) scale. This includes most structures of interest to science for the past 100 years, i.e., since the advent of non-optical probes such as diffraction methods and electron microscopy. Before this period, the optical microscope was the main tool for observation.

Diffraction methods include neutron scattering which has found wide use in the characterization of materials. Partial deuteration has made neutron scattering unique. Use of deuterated molecules in a non-deuterated environment is comparable to the staining method used in electron microscopy and helps enhance the contrast of particular structural features.

Small-angle neutron scattering (SANS) is a well-established characterization method for microstructure investigations in various materials. It can probe inhomogeneities in the nanometer scale. Since the construction of the first SANS instrument over 35 years ago, this technique has experienced a steady growth. SANS instruments are either reactor-based using monochromated neutron beams or time-of-flight instruments at pulsed neutron sources. SANS has had major impact in many fields of research including polymer science, complex fluids, biology, and materials science. This technique has actually become a "routine" analytic characterization method used even by non-experts.

This book is intended to help SANS users acquire (or brush up on) basic knowledge on the technique and its applications. Readers need not be experts in the various subjects covered here. Basic knowledge in areas like nuclear physics, basic chemistry, statistical mechanics and mathematics is of course helpful. The covered topics are organized into broad categories (parts) which are divided into chapters. Each chapter contains a number of related topics included as sections. Helpful questions (and answers) are included at the end of each chapter. The outlines of the various parts and the section titles are color coded; blue has been chosen for essential knowledge sections. Readers would benefit by first focusing on these sections.

After a brief review of basic neutron properties, the various methods of neutron production and various neutron sources are introduced first along with discussion of neutron flux. The major neutron sources are listed along with their overall characteristics. Production of cold neutrons (essential for SANS applications) is discussed along with description of cold neutron remoderators. Basic elements of neutron scattering follow. These include advantages and disadvantages of the technique, scattering lengths and cross sections, coherent/incoherent scattering contributions, and example calculations. This is followed by discussion of elastic/inelastic and coherent/incoherent neutron scattering. Elements of Quantum Mechanics are used to derive the scattering cross section.

The SANS technique is described next. SANS instrumentation is examined in no great detail focusing on the major components and pointing out differences between reactor-based and spallation source-based instruments. Neutron velocity selectors and area detectors are included here along with their calibration and discussion of their performance. SANS resolution and the various elements of instrumental smearing are described next. These include contributions from the instrument focusing geometry, wavelength spread and detector resolution as well as the effect of gravity on neutron trajectories. Instrumental resolution is also discussed when refractive optics (neutron lenses or prisms) are included.

Description of the various elements of SANS data correction and data reduction are included next. The main SANS data interpretation methods include standard plots, the use of empirical models and nonlinear least-squares fits to realistic models. Representative SANS data are presented. Elements of SANS data modeling include calculations of the radius of gyration, of the single-particle form factor and of inter-particle structure factors. The effect of polydispersity is also discussed. Since "most SANS spectra look alike", SANS is a heavily model-dependent method. The major theories used to interpret SANS data are discussed including the Random Phase Approximation (RPA) for polymer systems and the Ornstein-Zernike (OZ) equation for particulate scattering.

The major SANS research topics are covered in turn in a series of chapters. These various “parts” include: Polymers, Complex Fluids, Biology, and Other Topics that includes Materials Science. In each chapter, typical topics borrowed from the research efforts of this author are described at the tutorial level. The part on “SANS from Polymers” includes polymer solutions, polymer blends and copolymers. The Random Phase Approximation approach is described in detail and applied to realistic homogeneous polymer mixtures. The thermodynamics of phase separation are described for multi-component homogeneous polymer mixtures. The part on “SANS from Complex Fluids” includes a discussion of the phase diagram for micellar systems and contains chapters on ionic and nonionic “self-assembling systems”. The main scattering features include single-particle and inter-particle contributions. Material balance equations help in the understanding of some details of the probed structures. The part on “SANS in Biology” introduces elements of biology then covers representative basic topics such as a phospholipid membranes, the helix-to-coil transition in DNA and the structure of a protein complex.

The “Other SANS Topics” part is covered next. These include the effect of pressure or shear on nanoscale structures, solvation in mixed solvents, and molecular orientation of polymeric materials. SANS measurements involving in-situ pressure or in-situ shear have been the focus of research for many years. The effects of pressure on phase separation and miscibility are discussed. In-situ shear allows investigations of the rheology and structure simultaneously.

Chapters covering review of the literature in the four main SANS research areas have been included. These draw heavily from papers published (over the past seven years) from use of the NIST Center for Neutron Research.

Two other small-angle neutron scattering techniques are discussed in no-great detail in the part on "Even Lower SANS Scales". These are the Ultra small-angle (USANS) range probing structures as large as 20 microns and the merging VSANS technique (V is for very small-angle) which bridges the two probing ranges.

A gallery of interesting SANS data images is included. These images have been collected by this author over several years. They are included here in order to show the full richness of the SANS technique and for their esthetic value. Some brief concluding topics are covered along with two appendices; one on "Useful Mathematical Expressions" and the other on "Elements of Quantum Mechanics". These appendices gather material used throughout.

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