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Fe_3O_4 (magnetite) is a ferrimagnetic material that has been known to man for thousands of years, and was used to make the first magnetic compasses as early as the 8th century AD. In more recent times it has been greatly studied by the scientific community, with much effort going into investigation of the Verwey transition [1] and the associated structural changes [2]. Despite this, a definitive model for the low temperature structure has remained elusive.

In this study neutron total scattering data from a highly stoichiometric powder sample collected at 10K have been used to perform reverse Monte Carlo (RMC) refinements using the program RMCProfile [3]. These refinements produce large box atomic configurations consistent with experimental data on both local and average length scales. Atoms may be translated and magnetic moments swapped and rotated during the refinement, allowing both the atomic and magnetic structures to be refined.

The results and their implications are discussed within the context of charge ordering models and space group assignments produced by previous studies.

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Keywords: diffraction, magnetite, RMC

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Studying the 3-dimensional structure of proteins in solution by small-angle X-ray scattering

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Small-angle X-ray Scattering (SAXS) is an important method for the characterization of biological macromolecules. Usually, the structure of proteins is characterized by protein crystallography and NMR. However, the results obtained with these methods do often not reflect the protein structure and its behavior in the native state. With SAXS, the 3-dimensional structure of proteins in solution, i.e. under biological conditions can be determined. The method determines the size and shape of proteins and protein complexes. Furthermore, SAXS is very sensitive to any structural or size changes caused e.g. by external effects (changed temperature or pH, ligand binding, etc.).

The main challenge for measuring proteins in solution with laboratory SAXS systems is thelow contrast of these samples (contrast in SAXS is the difference in electron density between the nanoparticle and the matrix). Therefore, the design, collimation concept [1] and quality of the SAXS system is crucial for achieving excellent data quality at reasonable measurement times.

In this contribution we will show some examples of structural characterization of proteins in solution with the SAXSess mc² laboratory system. These examples include the determination of the overall protein size, of the folding state and of the 3-dimensional envelope describing the structure of the protein in solution. The determined solution structures can be well compared with structural data from protein crystallography.

SAXSess mc² is ideally suited for the analysis of e.g. low-concentrated protein solutions or drug delivery systems within minutes. Therefore, it can be used for time-resolved monitoring of structural changes. Furthermore, many samples can be screened by use of a fully integrated autosampler.

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Keywords: SAXS, Proteins, 3D-envelope

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New GI-SAXS capabilities for the home-lab

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Grazing-incidence small angle X-ray scattering (GI-SAXS) is an established technique for the characterization and analysis of nano-structured surfaces and thin films. Typical applications include thin organic films, quantum dot arrays or nanomaterials deposited and arranged on surfaces. However, GI-SAXS is mainly performed on dedicated synchrotron (GI-)SAXS beamlines due to the requirement of high primary beam intensity and parallel-beam requirements.

In this contribution we will present the new GI-SAXS stage for the SAXSess mc² laboratory small- and wide-angle X-ray scattering system. The precise GI-SAXS stage in conjunction with the SAXSess mc² system enables to perform GI-SAXS experiments even in the home laboratory. Samples with a nano-structured surface can be precisely tilted in order to perfectly obtain grazing incidence conditions. In addition, samples can be rotated in-plane in order to study the arrangement of the nanostructured surface. We will discuss technical aspects of the GI-SAXS stage and will present selected results of nanostructured thin film samples.

Keywords: GI-SAXS, grazing incidence, surface

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Non-crosslinking aggregation of DNA-functionalized nanoparticles

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Recently, it was discovered that DNA-functionalized nanoparticles assemble by hybridization in a non-crosslinking configuration. When single-stranded (ssDNA) molecules grafted on the nanoparticles hybridize to the complementary ones, the resulting nanoparticles, which are covered with double-stranded DNA (dsDNA), assemble immediately at high salt concentrations without molecular crosslinkng, while the nanoparticles with ssDNA molecules remain stable. The colloidal stability is very sensitive to the terminal base-pair of DNA duplex. When the tethered ssDNA hybridizes with a single-base mismatched DNA, the nanoparticles still disperse stably at the high salt concentrations. However, the mechanism of the non-crosslinking aggregation has not been understood yet. In this study, the non-crosslinking aggregation was explored from the structural point of view, using mainly small-angle X-ray scattering (SAXS). The structural characterization of the gold nanoparticles with ss- or dsDNA was performed, and the effects of DNA length, core size, and temperature on the non-crosslinking aggregation were also investigated.

Gold nanoparticles (ca. 5, 15 and 40 nm) were functionalized with ssDNA Here, the DNAs with various bases were used. The functionalized gold nanoparticles were suspended in 10 mM phosphate

buffer (PB) (pH 7.0) containing 0.1 M NaCl. Solution SAXS measurements were carried out at the BL45XU RIKEN Structural Biology Beamline I (wavelength, λ = 0.09 nm) of the SPring-8, Harima, Japan. Two-dimensional (2D) SAXS images were recorded with a CCD camera. For aggregation experiments, the complementary DNA and then NaCl were added to the suspension in such a way that the final concentrations of complementary DNA and NaCl were 0.5 μM and 1 M, respectively.

The non-crosslinked assembly has a crystalline structural order but less long-range ordering. The inter-surface distance between the particles increased with the length of dsDNA. From the experiments using the binary mixtures, it was unveiled that the DNA-functionalized particles with the same core size assemble together irrespective of tethered DNA. Furthermore, it was demonstrated that, for smaller core sizes or longer DNA lengths, their nanoparticles covered with full-matched dsDNA can disperse stably above a temperature which is lower than the melting temperature of the dsDNA. This may cause by steric stabilization of DNA molecules. It is considered that the attractive interaction in the non-crosslinking aggregation is attributable to van der Waals potential between core particles, and the contribution of the end-to-end stacking attraction between dsDNA might be little. As a plausible mechanism, the feature of non-crosslinking aggregation interaction could be characterized by steric repulsion stabilization due to entropic loss in conformation and mobility of DNA molecules.

Keywords: DNA, colloid, SAXS

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Further development of Brillouin zone database on the Bilbao Crystallographic Server

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The Bilbao Crystallographic Server (www.cryst.ehu.es) [1] is a web site offering online crystallographic programs and databases. The aim of this contribution is to announce the modifications and improvements of the Brillouin zone database available on the server. The database includes Brillouin zone figures (fig. 1) and tables of the wave-vectors for all space groups that form the background of the classification of the irreducible representations of all 230 space groups. The wave-vectors of reciprocal space are classified in terms of their symmetry described by the symmorphic space groups available in Volume A of *International Tables for Crystallography* (ITA). This is possible due to the isomorphism between the reciprocal space groups, describing the symmetry of the wave-vectors, and the symmorphic space groups in direct space [2]. In the database, the ITA-classification scheme is compared with the classification of the wave-vectors available in the tables of space-group representations by Cracknell, Davies, Miller and Love [3].

The recent modifications of the database have been focused on a general improvement and homogenization of the stored data and a clearer presentation of the Brillouin-zone figures. The wave-vector tables list for each space group the coordinates of the wave-vectors with respect to primitive (CDML) and conventional dual bases compared with the coordinates according to the ITA description. The Brillouin-zone figures for trigonal, hexagonal and rhombohedral space groups are redrawn applying a much more visual clinographic projection, which was also used for the rest of the space groups. The figures and tables for monoclinic space groups in *unique axis b* setting have been also included in the database.

In addition, we have developed a computer tool for the complete characterization of the wave-vectors: given the wave-vector coordinates referred to primitive or conventional dual bases, the program assigns the corresponding wave-vector symmetry type, specifies its CDML label, determines the little co-group of the wave-vector and generates the arms of the wave-vector star.

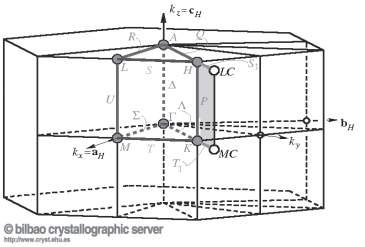

Figure 1: Brillouin-zone diagram for the space groups of the arithmetic crystal class 6/mmmP

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Keywords: brillouin zone, wave-vector symmetry, bilbao crystallographic server.

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The wwPDB common tool for deposition and annotation

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An early example of open data sharing, the PDB archive has provided the research community with information about the 3D structures of biological macromolecules for the past 40 years. From 7 structures in 1971 to more than 70,000 in 2010, PDB data are used by researchers in academia, government and industry, and educators.

The Worldwide Protein Data Bank (wwPDB; wwpdb.org) consists of four organizations from the USA, Europe, and Japan that together curate and disseminate the single, global PDB archive of biomacromolecular structures. The wwPDB is committed to using the highest standards of curation and validation to process experimental 3D biomolecular structure data.

The next 10 years will bring several challenges for this repository including significant increases in throughput and in the size and complexity of structures being deposited. In response to this challenge the wwPDB has initiated the *Common Deposition and Annotation Tool* project to produce the next generation of processes and tools. This new system will be used at all wwPDB sites to curate structural data produced by any combination of experimental techniques (X-ray, NMR, EM) to support the wwPDB goals of quality and dependability over the next 10 years. The new tools are designed to fulfill the goals of high standards of curation quality and dependability and to add value to depositors as they interactively deposit new structures. The