

Poster Sessions

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The duality of agarose gels as either impurity or impurity filter in protein crystal growth experiments has been discussed frequently. But, up today, we don't know if agarose gels operate as an impurity, as an impurity filter or both. Therefore a series of experiments have been performed using gelled solutions (low concentration agarose) and ungelled solutions to simultaneously study the effect of gels and impurities on the growth kinetics of biomacromolecule crystals. The growth of crystals from highly purified (99.99% purity) and commercial grade (98.5%) lysozyme was observed by Laser Confocal Microscopy combined with Differential Interference contrast Microscopy (LCM-DIM). Step velocities, 2D nucleation rates and normal growth rates were measured. These growth parameters were separately assessed for crystals growing from pure and commercial grade solutions. It was found that 2D nucleation rates are enhanced by the presence of gel fibers that act as heterogeneous nucleation sites. These results also show that the specific surface energy is similar for the gel fiber/crystal interface and for the gel fiber/solution interface. This is consistent with the observed incorporation of agarose fibers into the lysozyme crystal lattice and the small effect of gel fibers on step velocity for crystals growing from highly purified solution. The presence of agarose significantly modifies the step velocity in crystals growing from impure solutions, shifting these values closer to the velocities measured in purified solutions. This velocity increase corresponds to a 7 fold reduction in the concentration of adsorbed impurities at the crystal surface with respect to ungelled experiments and can be considered as direct evidence of the diffusive impurity filtering concept.

Keywords: protein crystal growth, agarose gel, impurity filtering

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Self-organized eutectic microstructures towards photonic crystals and metamaterials

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Eutectics are special materials which are both a MONOLITH and a MULTIPHASE MATERIAL.[1] They may find application in the field of photonic crystals and metamaterials.[2, 3] The eutectic microstructure can exhibit many geometrical forms. It can be regular-lamellar, regular-rod-like, irregular, complex regular, quasi-regular, broken-lamellar, spiral and globular. The most interesting from the point of view of photonic crystals would be the microstructures with regular shapes, i.e. lamellar and rod-like. For metamaterials applications the other shapes could be also of interest - for example the percolated structures (for giant dielectric constant); or the spiral one for chiral metamaterials. The general overview of the road of eutectics towards photonics as well as new experimental data will be presented.

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Keywords: eutectic crystallization, self-organization, photonic crystals and metamaterials

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Desktop Minstrel UVTM: A novel protein crystal monitoring automation system using UV fluorescence

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Identifying protein crystals in crystallization droplets has long been considered a challenging step in the field of protein crystallography. Although there are numerous automated crystallization robots readily available, none have been able to successfully monitor crystal growth by distinguishing protein crystals from non-protein crystals and detecting crystals from drops that are otherwise difficult to see with visible light. In order to fulfill this critical need, Rigaku has developed a novel protein crystal monitoring automation system, the Desktop Minstrel UVTM, which uses UV fluorescence microscopy. The system includes an ultraviolet microscope with at least one ultraviolet light emitting diode, providing illumination with the wavelength matching the absorption of the fluorescing amino acids, such as tryptophan. To greatly decrease photo-damage to the protein crystals, the fluorescing light illuminated on the sample is reduced to the minimum and is then digitally recorded by a camera with a CCD sensor. We have conducted crystallization experiments with various proteins in order to evaluate this system. The resulting UV images from these experiments clearly reveal the protein crystals from non-protein crystals, such as salts. In addition, this UV crystal monitoring system is built upon the platform of Rigaku's state-of-art imaging automation technology, the Desktop Minstrel, which makes the evaluation of a large number of crystallization experiments possible. The Desktop Minstrel UV enables researchers to accurately harvest protein crystals for data collection or design follow-up experiments.

Keywords: crystallization robots, imaging, UV fluorescence

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The microcapillary protein crystallization system

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The Microcapillary Protein Crystallization System (MPCS) embodies a new, semi-automated, microfluidic plug-based crystallization technology which allows researchers to conduct nanoliter-volume screening of crystallization conditions and to perform in-situ X-ray diffraction studies on crystals that form. The MPCS integrates formulation of crystallization cocktails with preparation of the crystallization experiment. Within microfluidic Teflon tubing or the microfluidic circuitry of a plastic CrystalCard, ca.10-20 nL volume droplets can be generated, each representing a traditional

microbatch-style crystallization experiment with a different chemical composition. The MPCS allows the researcher to use the entire protein sample in crystallization experiments for efficient exploration of crystallization phase space by combining sparse matrix with gradient screening in one comprehensive hybrid crystallization trial. Furthermore, individual crystallization optimization trials can be prepared using highly granular gradients of protein and optimization reagents such as precipitation agents, ligands, or cryo-protectants. The MPCS produces Diffraction-Ready crystals that can be removed from the Peel-Apart CrystalCard for traditional cryocooling and diffraction.

Keywords: microfluidic crystallization, nanovolume, *in-situ* X-ray diffraction”

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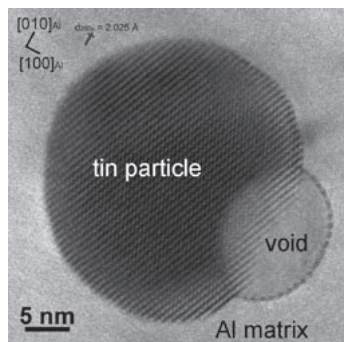
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Three-dimensional void-like defects associated with tin nano-particles in aluminium

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Defects, such as lattice defects (vacancies, dislocations, stacking faults) or extrinsic defects (solute atoms clusters) play a critical role in the nucleation and growth of precipitate phases in precipitation-strengthened aluminium alloys. Defects often act as heterogeneous nucleation sites for phases that nucleate with difficulty. Defects, vacancies in particular, may also influence or even control the kinetics of nucleation and growth. This work reports the finding and characterisation of three-dimensional defects commonly associated with tin nano-particles in aluminium. The shape, structure and composition of the defects and their surroundings were investigated using a variety of transmission electron microscopy imaging, diffraction and analytical techniques. The three-dimensional defects were deduced to contain a significant number of vacancies, hence their description as void-like. These void-like defects were found to occur in isolation at the interface between the tin precipitate and the aluminium matrix. An example of one such void observed at high magnification is shown; in this case tin can be seen to decorate the void-matrix interface.



Keywords: defects, transmission electron microscopy, aluminium alloys

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Microstructure of surface-tailored platinum nanocrystals

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Nanoscaled Pt-based materials have potential applications as electrocatalysts in fuel cells. Fundamental interest in the mechanisms of model reactions has led to the development of synthesis routes allowing a fine tuning of particle shape, mean size and size distribution. However, an effective control of the surface structure seems more important, as the majority of the reactions taking place in fuel cells are structure sensitive or site dependent (1). This fine control over surfaces is allowed by the water-in-oil or by the colloidal routes: single clean-surface nanocrystals with defined shapes and tailored percentages of {100} and {111} surface domains can be produced (2). In particular, the colloidal method allows cubes, octahedra, tetrahedra and cuboctahedra to be preferentially obtained (depending on the Pt precursors and the hydrogen bubbling time), whereas more rounded crystals are formed via water-in-oil. All nanocrystals are almost defect-free and nearly monodisperse, as confirmed by TEM and by X-ray diffraction Whole Powder Pattern Modelling (WPPM) (3). An analysis of the microstructure is proposed, based on the modelling of nanocrystal shape, size distribution, defects and surface effects, following the WPPM and the Debye equation approaches.

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Keywords: nanocrystals, platinum, microstructure

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Crystallite dimensions obtained with Rietveld refinement and Delaunay triangulation

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Crystallite dimensions and morphology of phases were obtained by refining crystalline structures with the Rietveld method. Of special interest was the case where average crystallites were modeled in reciprocal space with a linear combination of normalized spherical harmonics; the coefficients that weighted the harmonics' contribution were refined to fit the breadth of the diffraction peaks. The crystallite dimensions obtained in reciprocal space were used to calculate the corresponding ones in real space, generating a set of vertices that described crystallite surface. These vertices were used to generate a mesh of the surface using the Delaunay triangulation, which made possible to get crystallite surface area, and to generate a Delaunay tetrahedralization that was used to calculate crystallite volume. The density of each phase, determined from the Rietveld refinement, together with the determined volume were used to get crystallite mass and its specific surface area, which, for comparison, can be determined with other experimental techniques. Since in nanocrystalline materials peak breadth is mainly determined by crystallite size and microstrain, the aberrations of the diffractometer can be neglected, which is not the fall for microcrystalline materials.