2023

LASS carries out research in three areas: carbon based materials, computational material science and x-ray related methods. In the next paragraphs, our most important results are summarized.

Carbon based systems

We demonstrated the possibility of preparing graphene nanoribbons encapsulated in boron nitride nanotubes starting from room-temperature encapsulation of a liquid precursor. The room-temperature reaction precludes side products and results in pristine graphene nanoribbons upon annealing. The length of the nanoribbons depends on the annealing temperature and the type of nanoribbon on the diameter of the boron nitride nanotubes. We performed structural characterization by transmission electron microscopy and spectrosopic characterization by Raman spectroscopy. Graphene nanoribbons are important for nanoelectronics, their electronic structure depending on their length and conformation. [1]

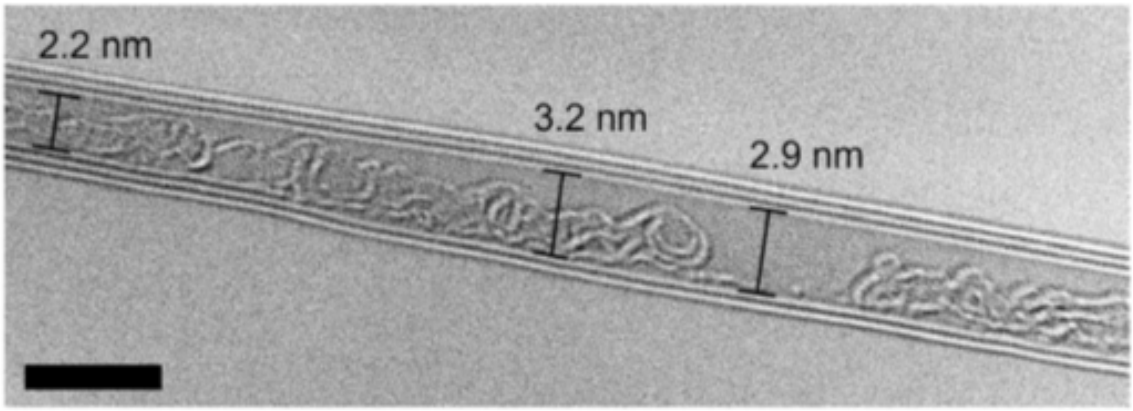

Fig. 1 Carbon structures formed inside a BNNT. In the small-diameter part on the left, the characteristic twist of GNRs is observed; in the wider areas in the middle, the encapsulated material forms more disordered structures; and on the right there is an empty volume [1]

We also followed the transformation of amorphous calcium carbonate to calcite by infrared spectroscopy at different temperatures. The transformation occurs in the presence of air, its rate increasing with temperature. We compared the data with x-ray diffraction and calorimetry measurements. The details of the transformations between various calcium carbonate polymorphs are of paleoclimatological importance. [2]

Computational Materials Science

Phase-field modelling, which is a standard materials science tool, was used to study biomineralization during nacre formation in mollusk shells. Mollusks, as well as many other living organisms, can shape mineral crystals into unconventional morphologies and to assemble them into complex functional mineral–organic structures. Our modeling efforts helped us to identify the key physical parameters that govern this process. The results support the hypothesis that the control over mineral morphogenesis in mineralized tissues happens via regulating the physicochemical environment, in which biomineralization occurs: the organic content manipulates the geometric and thermodynamic boundary conditions, which in turn, determine the process of growth and the form of the biomineral phase. The approach developed here has the potential of providing explicit guidelines for the morphogenetic control of synthetically formed composite materials [3].

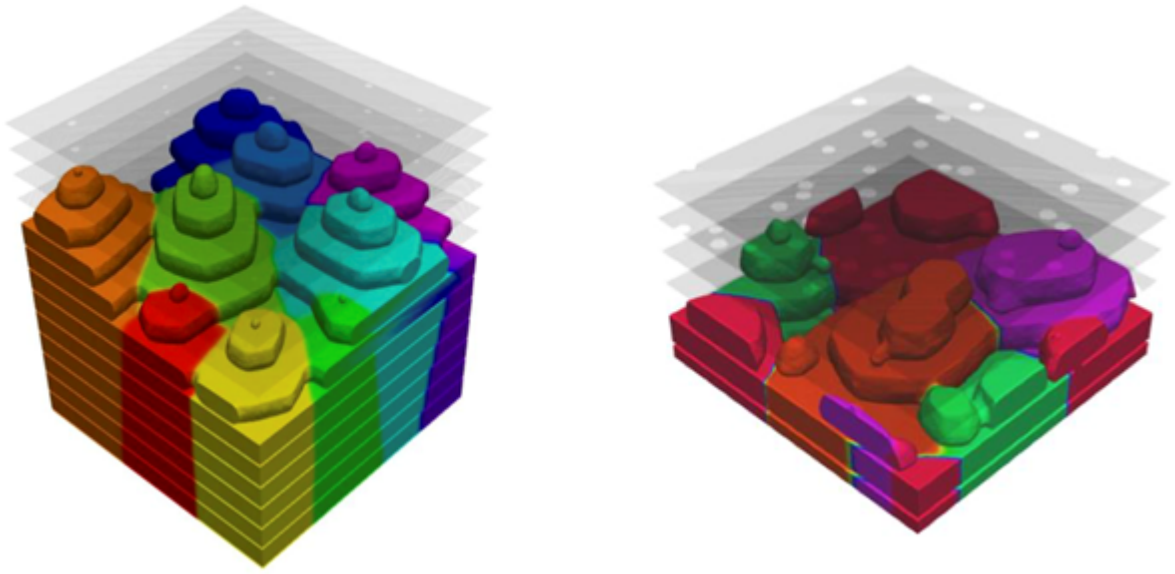

Fig. 1: Phase-field modeling of the formation of columnar (left) vs. sheet nacre (right). In both cases, crystallization of aragonite tablets (shown in colors) happens in thin layers between organic sheets (gray). The markedly different microstructures are results of the different position of holes via which crystallization can proceed to the next layer. In gastropods, these holes are aligned, leading to an organized, columnar structure (left), while in bivalves they are random, resulting in a more disordered structure (right).

…

X-ray related methods

In the fall of 2022, we have done a successful experiment on single pulse imaging at the European XFEL. In 2023 we worked on the evaluation of the Kossel patterns taken on GaAs and GaP during the experimental run. We prepared an article, which was accepted in Nature Communications. The publication will appear at the beginning of 2024. In this article, we demonstrate that *ab initio* 3D structure determination is possible within 25 fs, which is many orders of magnitude shorter than any other structure determination done so far. These measurements open the way for the study of very fast non-repeatable processes such as phase transitions at extremely non-ambient conditions; high pressure, high magnetic field, high temperature. During the same experimental run, we showed that the Jungfrau detector 16 memory cell mode works at the XFEL conditions [4]. This type of data collection will be useful for time resolved structural studies on short time scales.

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2022

LASS carries out research in three areas: carbon based materials, computational material science and x-ray related methods. In the next paragraphs, our most important results are summarized.

Carbon based systems

We extended our near-field scattering microscopy measurements to the detection of plasmon polaritons in metallic carbon nanotubes. One-dimensional Luttinger liquid plasmons create an interference pattern that can be mapped with the same AFM tip that created the electric field to launch the plasmons. The propagating plasmon polaritons are strongly coupled to the substrate phonons, leading to the disappearance of the interference pattern at the phonon frequency. We demonstrated this effect on a carbon nanotube in contact with a SiO_2/hexagonal boron nitride substrate and estimated the coupling being in the ultrastrong regime [1]. The coupling with boron nitride is confined to the 6 nm thick layer without any influence from the underlying silica, thus paving the way for the precise detection of molecular monolayers.

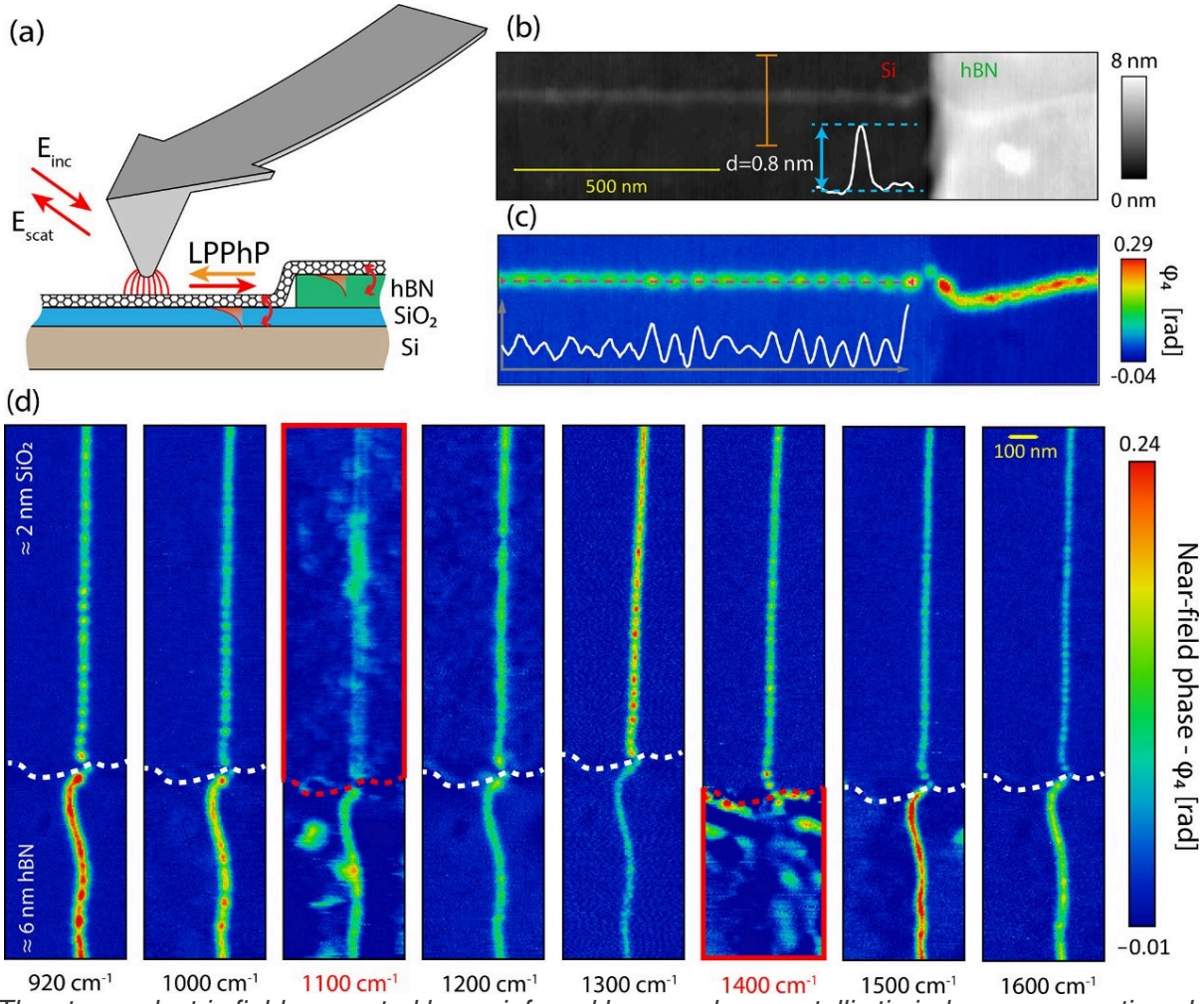

Fig. 1 The strong electric field generated by an infrared laser under a metallic tip induces propagating plasmons in a metallic nanotube deposited on a silica/hBN substrate (a). The topography (b) and optical contrast (c) of a metallic carbon nanotube showing different interference patterns on the two substrates. Sweeping the excitation frequency causes the interference patterns disappear at the phonon frequencies (1100 cm^{-1} for silica, 1400 cm^{-1} for hBN). [1]

Computational Materials Science, biomineralization

Besides studying the point-to-set correlation in glasses, and multistep nucleation using the Phase-Field Crystal theory [2, 3], and problems in biomineralization, we investigated the melting of 2D lamellar and 3D rod structures in a model eutectic system using multi-phase-field simulations [4]. Inverting the pulling speed of the sample in the standard directional solidification setup, steady state melting profiles were obtained as the long-time solutions in the minimal representative domains of the periodic structures. Simulations were performed using different volume fractions of the initial solid phases, different values of the temperature gradient, and different widths of the simulation domain. It was found that melting occurs with a nearly flat interface if the average composition of the initial solid structure is equal to the eutectic composition. If the volume fraction of the solid phases is changed and the average composition becomes off-eutectic, the melting positions of the phases decouple (Fig. 2): the phase of sub-eutectic amount will melt near the eutectic temperature, while the phase of super-eutectic amount will melt near its liquidus temperature corresponding to the off-eutectic mean composition. We observed two kinds of

instabilities in the regime of non-planar melting both in 2D and 3D: (a) Increasing the lamellar spacing, oscillations may appear around the trijunction and along the phase boundaries. (b) Decreasing the lamellar spacing and increasing the pulling speed, the lamellae/rods of the phase that protrude deeper in the melt become thinner and may eventually break up into a series of small spherical particles before melting completely.

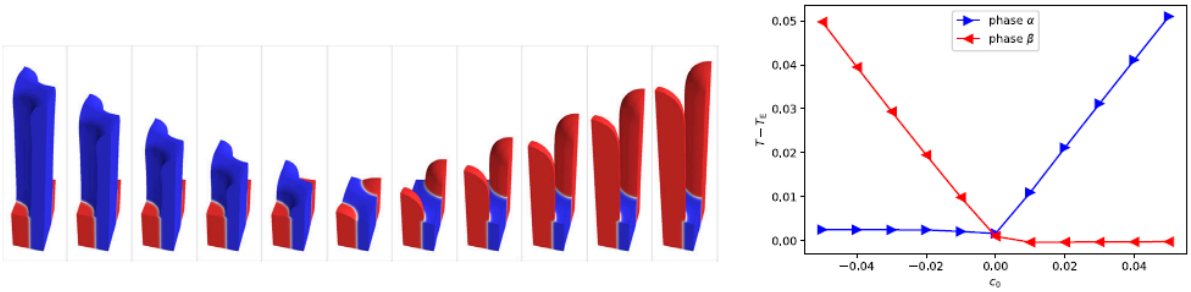

Fig. 2: Melting of 3D rod structures at different solid compositions. (a) Steady state morphologies obtained when melting a solid with different rod diameters that correspond to c_0 = –0.05, –0.04, ... , 0.05. The solid-liquid interface is shown. Blue and red denote the α and β phases. (b) Temperature values taken at midpoints of the α and β domains.

X-ray related methods
In the fall of 2022, we were granted beamtime at the European XFEL. In the first part of the year, we prepared for this measurement by sample preparation and modelcalculations. Based on these we have done successful experiments on single pulse imaging. We measured Kossel line patterns by single XFEL pulses on several materials (NiO, GaAs, GaP, Ge). This demonstrates that 3D structure determination is possible within 30 fs. These measurments open the way for the study of very fast non-repeatable processes such as phase transitions at extremely non-ambient conditions, high pressure, high magnetic field, high temperature. The evaluation of these measurements are under way.

2021

LASS carries out research in three areas: carbon based materials, computational material science and x-ray related methods. In the last year we have reached significant results in all of these fields.

Carbon based systems: Our newest results were achieved by using near-field infrared microspectroscopy on fullerene molecules encapsulated in boron nitride nanotubes. By mapping the near-filed scattering at the fullerene vibrational modes we could detect the molecules with a sensitivity limit of less than 200 molecules. We also followed the photopolymerization reaction of the encapsulated species and identified the products based on their infrared spectra. We conclude that the detection of the scattering is made possible by coupling of the molecular vibrations to phonon polariton modes of the nanotube. [1]

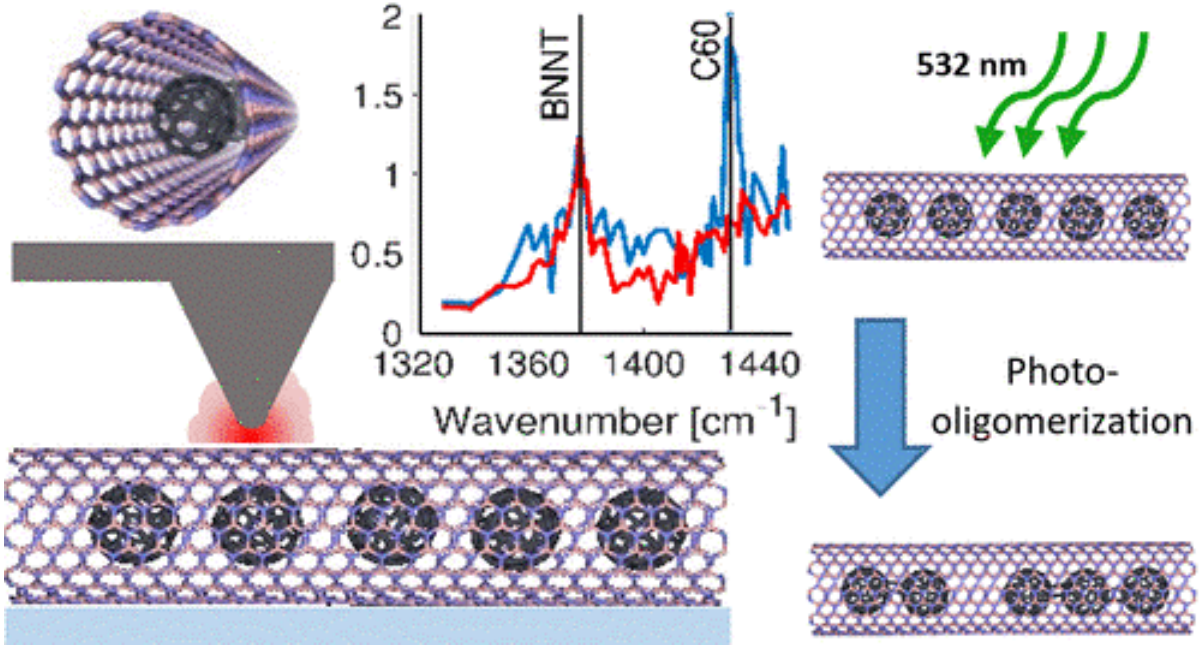

Fig. 1. Fullerene molecules are detected by illumination of an AFM tip by infrared laser light, with spatial resolution of ~ 20 nm. Visible light induces polymerization reactions in the sample, and the products can be identified based on their vibrational modes. [1]

Computational Materials Science, biomineralization: While biological crystallization processes have been studied on the microscale extensively, there is a general lack of models addressing the mesoscale aspects of such phenomena. We investigated whether the phase-field theory developed in materials science for describing complex polycrystalline structures on the mesoscale can be meaningfully adapted to model crystallization in biological systems [2]. We demonstrated the abilities of the phase-field technique by modeling a range of microstructures observed in mollusk shells and coral skeletons (Fig. 1), including granular, prismatic, sheet/columnar nacre, and sprinkled spherulitic structures. We also compared two possible micromechanisms of calcification: the classical route, via ion-by-ion addition from a fluid state, and a nonclassical route, crystallization of an amorphous precursor deposited at the solidification front. We have shown that with an appropriate choice of the model parameters, microstructures similar to those found in biomineralized systems can be obtained along both routes, though the time-scale of the nonclassical route appears to be more realistic. The resemblance of the simulated and natural biominerals suggests that, underneath the immense biological complexity observed in living organisms, the underlying design principles for biological structures may be understood with simple math and simulated by phase-field theory.

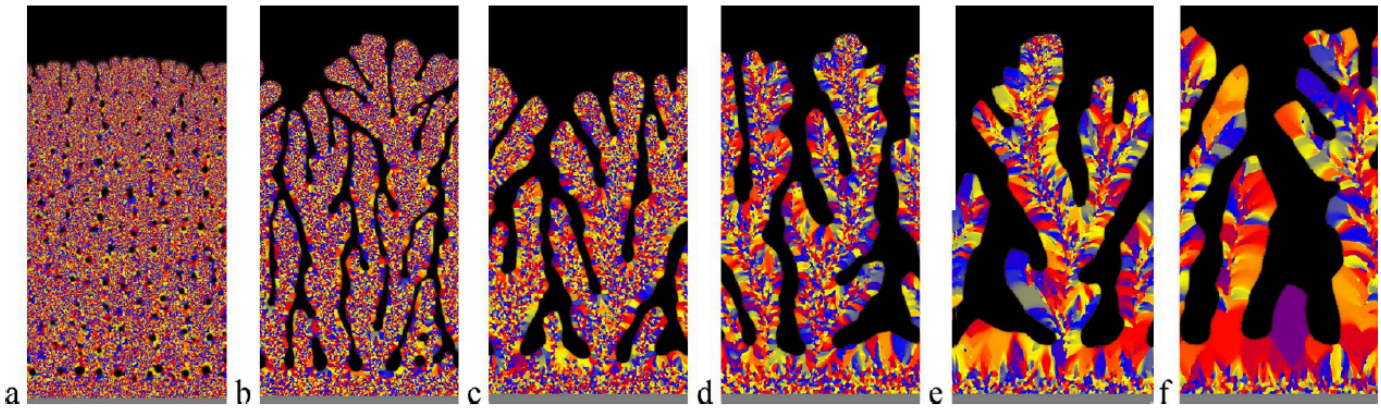

Fig. 2: Phase-field predictions for coral skeletons: Morphology and microstructure vs. temperature as predicted by theory [2]. Orientation maps are shown (different colors correspond to different crystallographic orientations, the liquid phase is black). The temperature increases from left to right. Note the decreasing amount of sprinkles (tiny crystallites) with increasing temperature.

X-ray related methods:
Our research concentrated on the evaluation of X-ray Free Electron based single molecule imaging experiments. We developed a simple and efficient method, the correlation maximization (CM) for orienting single particle diffraction patterns. In a recent work we compared our method to the EMC (expansion maximization and compression method) [3]. We found that at higher XFEL pulse fluences both the EMC and the CM algorithms give reliable results. However, the 3D intensity volume reconstructed by the CM method is more accurate than the one by the EMC method. For lower incident XFEL fluences, the CM method fails, while the more sophisticated EMC method can still converge to a meaningful solution.

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2020

LASS carries out research in three areas: carbon based materials, computational material science and x-ray related methods. In the last year we have reached significant results in all of these fields.

Carbon based systems.
One of the unique applications of carbon nanotubes is their use as nanocontainers for various encapsulated species. We could succesfully encapsulate sexithiophene molecules in single-walled carbon nanotubes using two different methods: sublimation filling at 400 °C and nanoextraction from supercritical carbon dioxide at 50 °C. We observed thermally induced polymerization during sublimation filling, which increased the electronic conjugation length of the chains inside the nanotubes. Although the conjugation length in the thiophene chain is increasing upon heating, no Raman signatures of graphene nanoribbons are observed [1]. These results complement our earlier studies, in which metal nanoclusters were encapsulated in the nanotubes.

An other type of the carbon based systems are fullerene cocrystals. Based on the analysis of the supramolecular interactions in several high-symmetry fullerene cocrystals, a consistent model has been worked out to predict the formation and stability of similar high symmetry molecular crystals [2]. In one of the cocrystal built from metallofullerenes and highly energetic cubanes, an anomalous, irreversible, pressure-driven lattice expansion has been detected by applying high pressure. Based on detailed theoretical and experimental study of this system, a new strategy was suggested for the creation of composite materials with similar negative volume compressibilities with exciting potential applications in various fields [3].

Computational Materials Science: In European collaboration, we investigated microstructure evolution during laser additive manufacturing (LAM) of hyperfine eutectic alloys in the Fe-Ti system, with the aim of designing materials for application in supercharger propeller (Bosch) and cloth cutting device (Procter and Gamble). Modelling of the microstructure was performed in the Wigner RCP using an orientation field-based phase field model, under heating and cooling conditions typical to LAM [4]. A layered structure consisting of alternating thin globular layers and extended lamellar domains was observed in the experiments. Its origin was explained in terms of a repeated remelting, nucleation, and growth sequence taking place after adding a new layer during LAM, as indicated in Figs. 1 and 2.

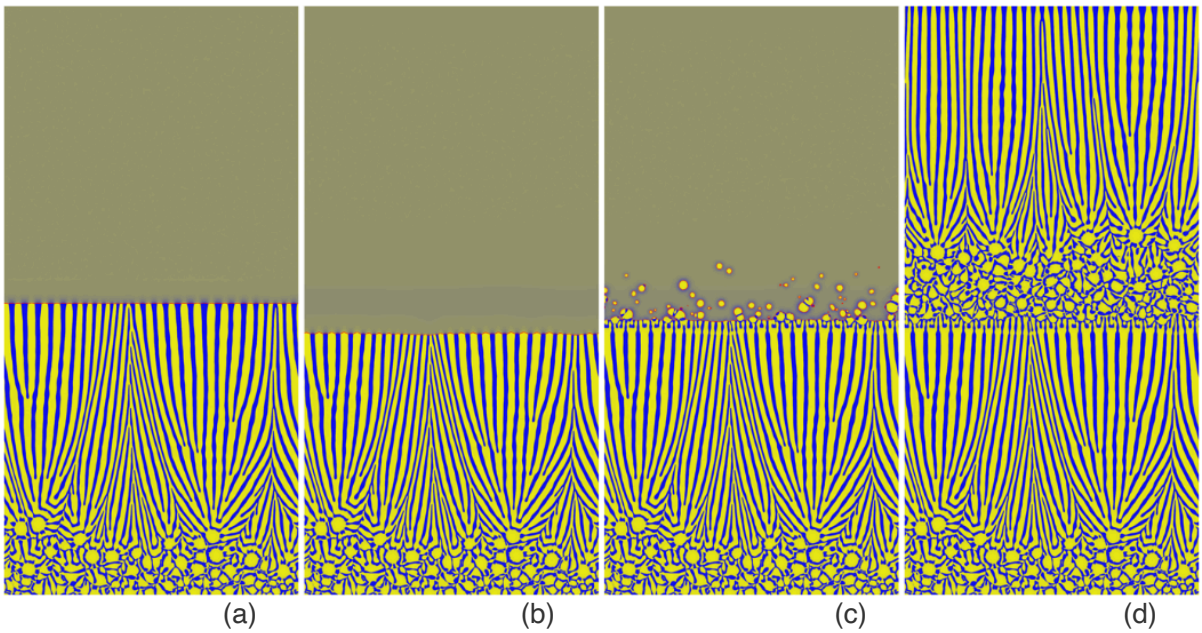

Fig. 1: *Snapshots of the composition map from the phase-field simulation of melting and solidification during a full heating/cooling cycle of laser additive manufacturing. Time elapses left to the right showing (a) the lamellar eutectic structure from the previous cycle and freshly melted powder on the top, (b) the remelting the topmost part of the lamellar structure during heating, (c) the epitaxial growth of the lamellae and the nucleation of the primary phase ahead and (d) the subsequent growth of elongated eutectic grains during cooling. At this point, the simulation domain is stepped up by half the sample height and a new heating/cooling cycle can be started with a configuration similar to (a).*

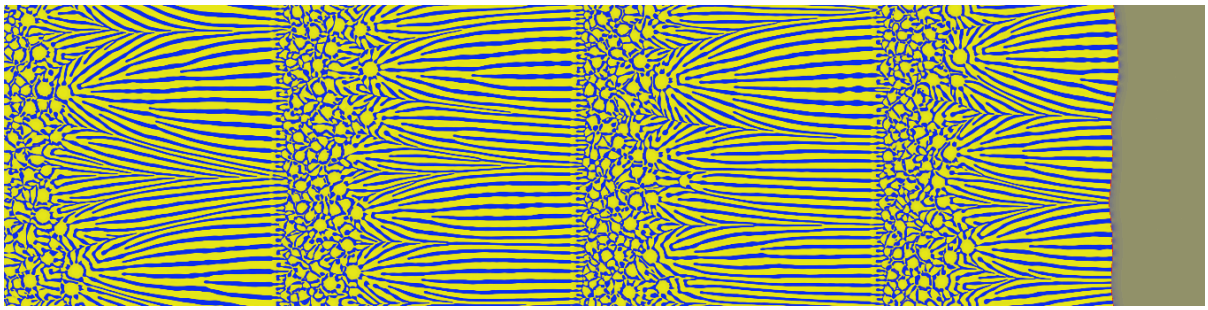

Fig. 2: *Composition map of the layered microstructure seen in LAM-produced eutectic alloys as predicted by the phase-field theory.*

X-ray related methods

Our research concentrated on the evaluation of Kossel patterns, which contain full crystallographic information on the sample, allowing unambiguous determination of the atomic structure. An autoindexing program was developed [5]. It allows the automatic indexing of the Kossel lines without preliminary knowledge of the crystal lattice. This work is essential for single pulse imaging experiments planed at XFEL-s, which will facilitate the measurement of various short lived phases at extremely non-ambient conditions.

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[5] https://doi.org/10.1107/S1600576720014892

2019

LASS carries out research in three areas: carbon based materials, computational material science and x-ray related methods. In the last year, we have reached significant results in all of these fields.

Carbon based systems. — As a new category of solids, crystalline materials constructed with amorphous building blocks expand the structure categorization of solids. New amorphous carbon clusters are found by compressing C8H8/C60 cocrystals, in which the highly energetic cubane (C8H8) exhibits unusual role. The significant role of C8H8 is to stabilize the boundary interactions of the highly compressed or collapsed C60 clusters, which preserve their long-range ordered arrangement up to 45 GPa. With increasing time and pressure, the gradual random bonding between C8H8 and carbon clusters, -due to “energy release” of highly compressed cubane, leads to the loss of the ability of C8H8 to stabilize the carbon cluster arrangement. Thus, a transition from short-range disorder to long-range disorder (amorphization) occurs. The spontaneous bonding reconstruction most likely results in a 3D network in the material, which can create ring cracks on diamond anvils.

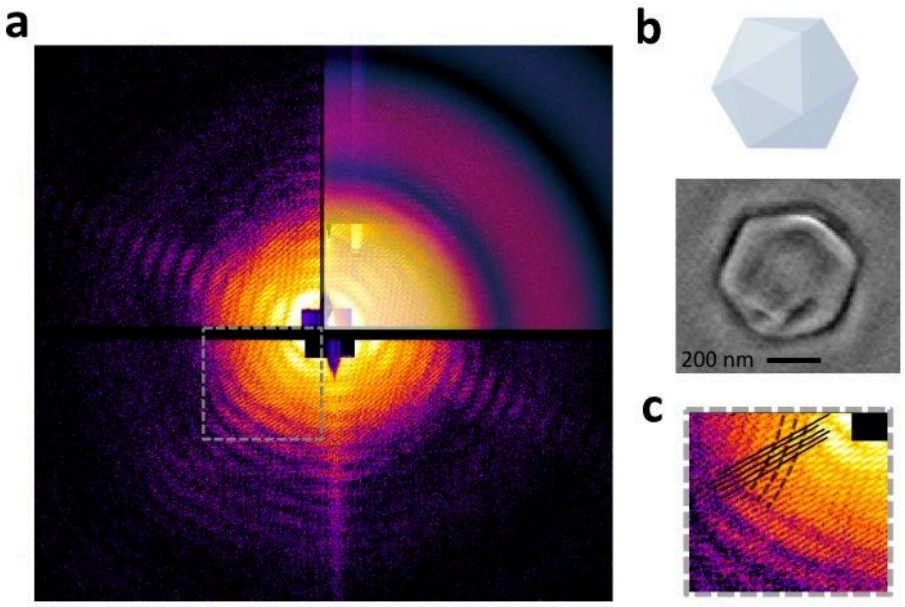

Figure 1. *(a) X-ray diffraction pattern of 3 particles, 2 Ar clusters and a Mimi virus. The diffraction pattern can be analogously analized as a hologram. (b) The icosahedral envelop of the mimi virus and its 2D SEM projection. (c) Enlarged part of (a) showing two fine line patterns, corresponding to the 3 particles in the beam.*

Computational material science. — During biomineralization, such as during the formation of bones, teeth, or mollusc shells and coral skeletons, hierarchically structured organic-inorganic composites of unique properties form, where the unique properties originate from their microstructure. We used traditional computational materials science tools to model the formation of these complex microstructures. In collaboration with German experimental scientists, we have reported a spectacular agreement between the microstructure of mollusc shells determined by experimental methods (including electron microscopy, electron back-scattering diffraction (EBSD), and X-ray micro-tomography) and the microstructure predicted by the phase-field theory. Such a complex structure was addressed for the first time by phase-field modelling, which may open the way for the modelling of even more complex biomineralization processes.

X-ray related methods. — Our research concentrated on X-ray free-electron laser (XFEL) related experiments. We took part in a holographic experiment on free-flying nanoparticles. We showed that structural information of individual nanoparticles could be obtained using a single XFEL pulse only.

Further, we gave general guidelines for the treatment of experimental data from coherent diffractive imaging and incorporated particle symmetry into the orientation determination in single-particle imaging (Fig. 1).

2018

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Carbon based systems.

As a new category of solids, crystalline materials constructed with amorphous building blocks expand the structure categorization of solids. New amorphous carbon clusters are found by compressing C_8H_8/C_{60} cocrystals, in which the highly energetic cubane (C_8H_8) exhibits unusual role. The significant role of C_8H_8 is to stabilize the boundary interactions of the highly compressed or collapsed C_{60} clusters, which preserves their long-range ordered arrangement up to 45 GPa. With increasing time and pressure, the gradual random bonding between C_8H_8 and carbon clusters, -due to “energy release” of highly compressed cubane-, leads to the loss of the ability of C_8H_8 to stabilize the carbon cluster arrangement. Thus, a transition from short-range disorder to long-range disorder (amorphization) occurs. The spontaneous bonding reconstruction most likely results in a 3D network in the material, which can create ring cracks on diamond anvils.

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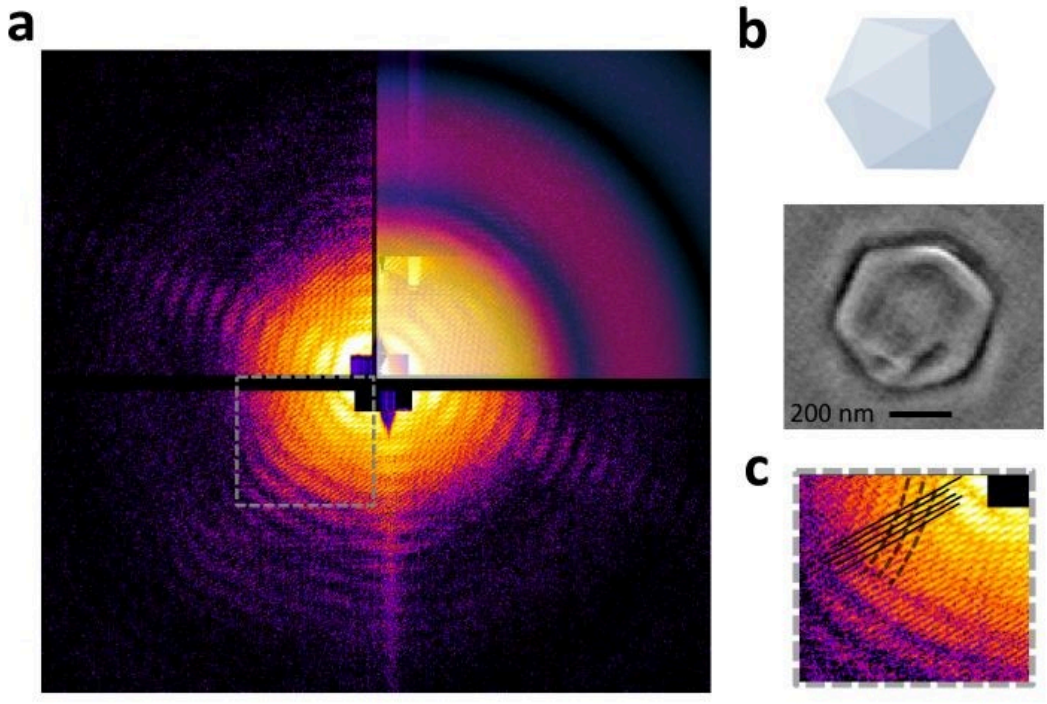

(a) X-ray diffraction pattern of 3 particles, 2 Ar clusters and a Mimi virus. The diffraction pattern can be analogously analized as a hologram. (b) The icosahedral envelop of the mimi virus and its 2D SEM projection. (c) Enlarged part of (a) showing two fine line patterns, corresponding to the 3 particles in the beam.

Further, we gave general guidelines for the treatment of experimental data from coherent diffractive imaging and incorporated particle symmetry into orientation determination in single-particle imaging.

2017

LASS carries out research in three areas: carbon based materials, the theory of phase transformations and x-ray-related methods. In the last year we have reached significant results in all of these fields.

Carbon based systems

Lately, various carbon based materials became the center of intensive research. Earlier we concentrated on fullerenes and related compounds. Recently, metal organic framework materials (MOF), carbon nanotubes and nanotube-based hybrid systems are our center of interest.

Metal-organic frameworks. — Metal organic frameworks are coordination polymers with high porosity. These crystalline, high-symmetry materials consist of metal-containing nodes and rigid organic linkers. Formerly, we developed a new MOF family with Zn-based secondary building units (SBUs) and 1,4-cubanedicarboxylate linkers. This year, we started a new family of MOF-s with the same Zn-containing nodes, but with a previously unknown spiroheptane-dicarboxylate linker. We prepared the organic precursor in a racemic form and demonstrated the formation of the new MOFs. We made ready the raw material for chromatographic separation of the enantiomers. The goal of the experiment is the preparation of chiral MOFs, suitable for separation of various racemic mixtures.

Infrared spectroscopy on carbon based systems. — We changed the focus of our research to include two new topics: near-field infrared spectroscopy and microscopy of various nanotubes and optoelectronic properties of organic perovskite-based solar cell materials. We succeeded in determining the semiconducting or metallic character of individual carbon nanotubes below 10 nm in diameter. In the case of boron nitride nanotubes, we mapped the defect distribution with a spatial resolution of a few nanometers.

We combined methylamine lead iodide perovskite with carbon nanotubes to obtain hybrid structures for possible application as photovoltaic devices. We proved the charge transfer from the perovskite to the carbon nanotube layer upon illumination. These observations may lead to new solar cells with the perovskite as active layer and the carbon nanotubes as hole-transporting layer.

Theory of phase transformations

Hydrodynamic theory of freezing – Nucleation and polycrystalline growth. — Structural aspects of crystal nucleation in undercooled liquids are explored using a nonlinear hydrodynamic theory of crystallization we proposed recently, which is based on combining fluctuating hydrodynamics with the phase-field crystal (PFC) theory. We have shown that in our hydrodynamic approach not only homogeneous and heterogeneous nucleation processes are accessible, but also growth front nucleation, which leads to the formation of new (differently oriented) grains at the solid-liquid front in highly undercooled systems. Formation of dislocations at the solid-liquid interface and interference of density waves ahead of the crystallization front are responsible for the appearance of the new orientations at the growth front that lead to spherulite-like nanostructures (Fig. 1).

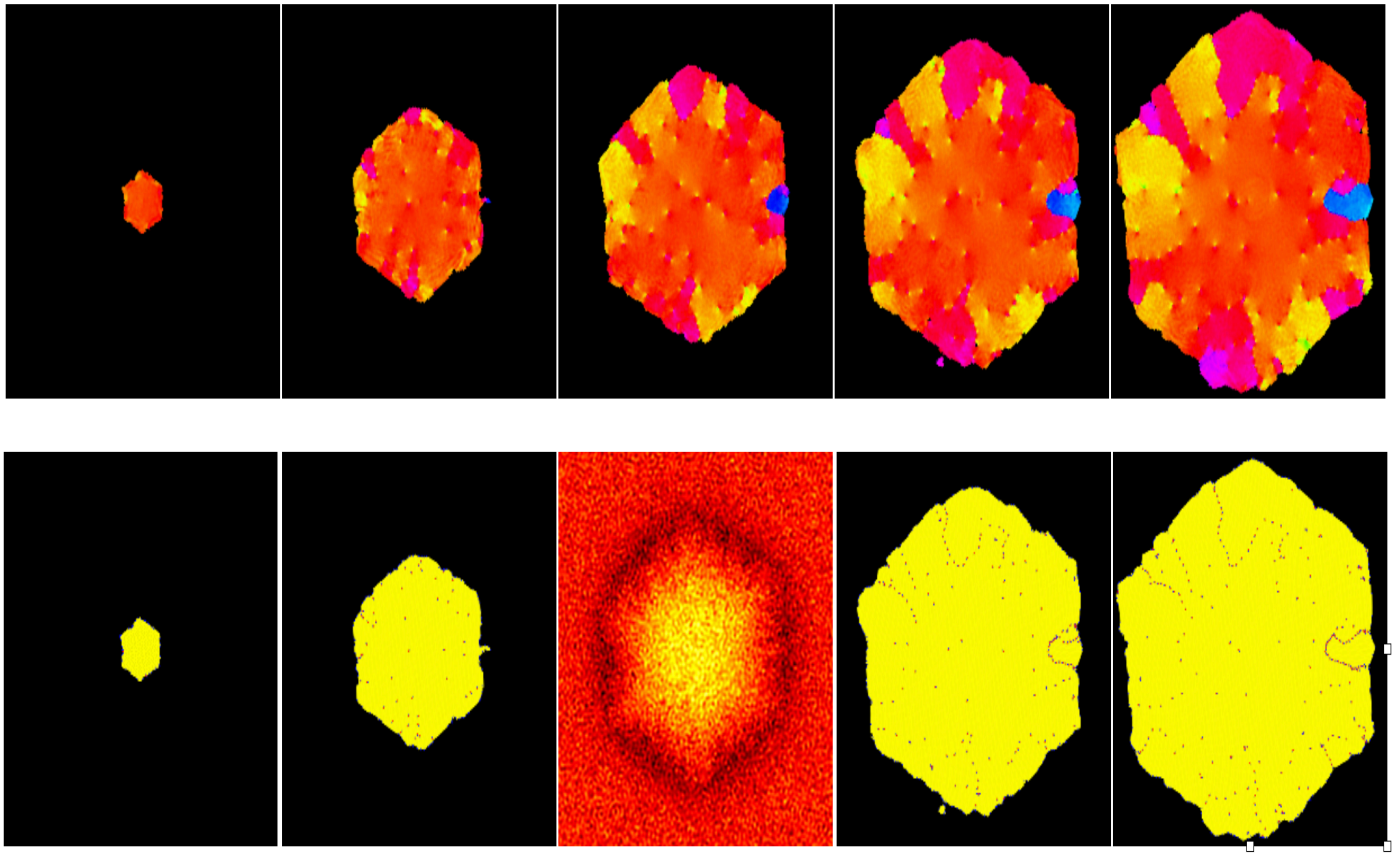

Figure 1. *Polycrystalline growth in the hydrodynamic model of freezing. Snapshots of the orientation field (upper row), the Voronoi map (bottom row), and coarse-grained density (bottom row central panel: lighter colour denotes higher density) taken at dimensionless times t = 900, 2100, 2900, 3400, and 3900 are shown. Note the spatial variation of the orientation field due to the dislocations shown as red-blue pairs of dots (atoms of 7 and 5 neighbours) in the Voronoi map, and the small crystallite formed close to the interface in the 4th panel from the left. This indicates two mechanisms for growth front nucleation: (i) nucleation of dislocations at the interface, and (ii) crystal nucleation ahead of the growth front.*

Grain coarsening in two-dimensional phase-field models with orientation field. — Contradictory results were published regarding the form of the long-time grain size distribution (LGSD) that characterizes grain coarsening in two-dimensional systems: While experiments and the PFC model indicate a log-normal distribution, other works including studies based on phase-field simulations that rely on coarse-grained fields, like the multi-phase-field and orientation field (OF) models, yield significantly different distributions. We investigated this problem, and demonstrated for the OF models that an insufficient resolution of the small-angle grain boundaries leads to a log-normal distribution close to those seen in the experiments. Our work also indicates that the LGSD is critically sensitive to the details of the evaluation, and raises the possibility that the differences among the LGSD results from different sources originate from differences in the detection of small-angle grain boundaries.

Topological defects in two-dimensional orientation-field models. — In 2D, a continuous scalar field is used to represent crystallographic orientation. The respective order parameter space is the unit circle, which is not simply connected. This property has important consequences for the multigrain structures: (i) trijunctions may be singular; (ii) for each pair of grains, there exist two different interfacial solutions that cannot be continuously transformed to each another; (iii) if both solutions appear along a grain boundary, a topologically stable singular point defect forms between them. While (i) can be interpreted in the classical picture of grain boundaries, (ii) and, therefore, (iii) cannot. To overcome these problems, we proposed two solutions. The first is based on a three-component unit vector field, while in the second we utilize a two-component vector field with an additional potential. In both cases, the additional degree of freedom makes the order parameter space simply connected, which removes the topological stability of these defect.

X-ray-related methods

We have continued our studies on structure determination by inside x-ray sources. We have carried out a series of experiments at ESRF, and measured atomic resolution holograms and also Kossel line patterns. The atoms which we used as point sources were exited by a very intense, synchrotron-generated focused X-ray beam. The diffraction patterns and the holograms were detected by a new 2D position sensitive detector allowing the

collection of higher quality data then in earlier measurements. The evaluation of the data is under way. This type of measurements open the way to single-pulse structure determination at X-ray free-electron lasers.