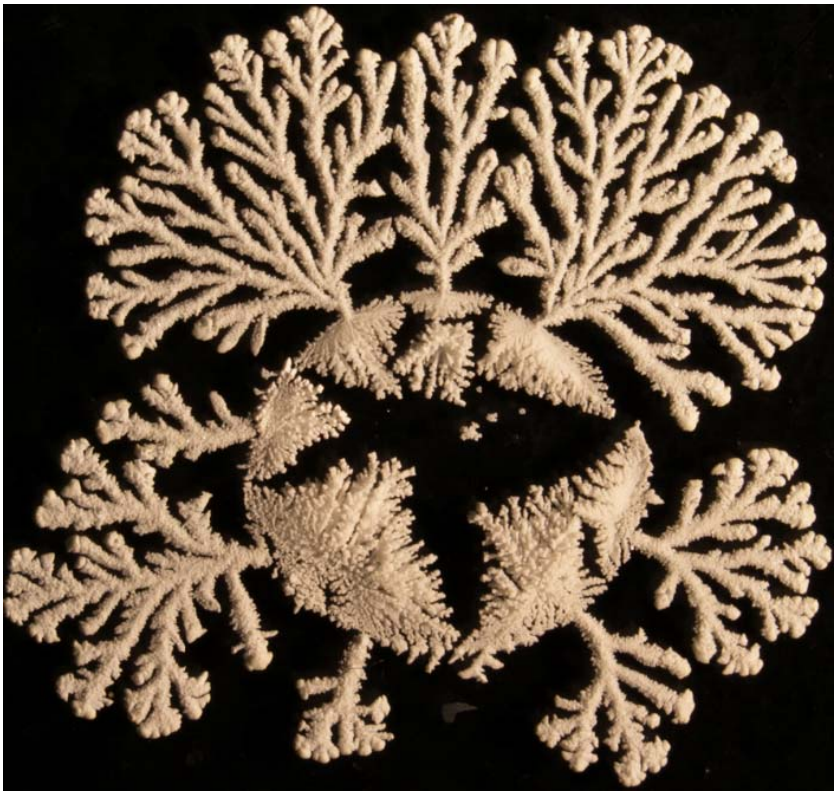
FACET

DACG NEWSLETTER

dutch association for crystal growth
DACG
NVKG
nederlandse vereniging voor kristalgroei



December 2017
issue 2

FACET
Newsletter of the Dutch Association for Crystal Growth (DACG), section of the KNCV and the NNV.

Editor:
dr. Arie van Houselt

Editorial office:
dr. A. van Houselt
Physics of interfaces & Nanomaterials
Universiteit of Twente
P.O. Box 217
7500 AE Enschede
The Netherlands
tel (053) 4893929 (3147)
fax (053) 4891101
A.vanHouselt@utwente.nl

CONTENT: **PAGE:**

DACG Secretary

prof.dr. Antoine van der Heijden
Leeghwaterstraat 39
2628 CB Delft

DACG board

dr. Hans te Nijenhuis (PANalytical)	chairman
prof.dr. Antoine van der Heijden (TUD)	secretary
dr. Pieter Vonk (DSM)	treasurer
dr. Marketta Uusi-Penttilä (Aspen Oss)	FACET
dr. Hugo Meekes (RU)	WWW

Cover figure

The creeping pattern which evolves from the evaporation of a saturated sodium chloride solution containing 1% (w/w) ferrocyanide as an additive. This evaporation-driven extension of crystallites from the solution boundary of sodium chloride has been found to be much enhanced by the presence of some specific additives.
The figure is provided by Ellie Townsend from the Solid state Chemistry group at the Radboud University in Nijmegen.

From the editor

In this second issue of the FACET we offer you a variety of crystal growth related information, ranging from an illustrated impressions from the DACG spring meeting in Delft on April 7th and DACG annual meeting on October 6th, at the site of JNJ in Beerse, Belgium, to an announcement of KNCV Piet Bennema Prize for Crystal Growth.

Our network of correspondents is steadily growing. The idea behind this is to get information from all the groups active in the field of crystal growth. Currently we have correspondents at the universities of Delft, Leiden, Wageningen, Nijmegen, Twente, Eindhoven and Amsterdam. Suggestions for correspondents from the missing ones are still most welcome! The correspondents will provide us with news on the research highlights, conference announcements and visits, and the new PhD theses from their groups. In the first stage of setting up the network, we focused on the academic groups. Next on the agenda is to get connected with our industrial members.

As always, it is easy to contribute to the Facet: Contributions may be delivered by mail, fax, e-mail, or telephone. Your ideas are welcome, please notify the new editor: Marketta Uusi-Penttilä

Arie van Houselt

Announcement

Innovation in Materials Characterization Award for Joost Frenken

On the MRS Spring Meeting in April, our former chairman Joost Frenken was awarded with the *Innovation in Materials Characterization Award* for his work on the development of scanning tunneling microscopes capable of imaging at industrially relevant conditions. Joost, congratulations from the DACG!

DACG Spring Meeting, Delft, April 7th, 2017

On April 7, 2017, the DACG organized its annual Spring Meeting at the section Intensified Reaction and Separation Systems (IRS), within the dept. Process & Energy of the Delft University of Technology. The theme of the symposium was “Crystallization and Process Intensification”. Historically, the DACG spring meeting provides a platform especially for PhD students to present their research.

The participants at the spring meeting in Delft.

This time, also two “nestors” in the field of crystallization were invited to give a presentation, namely **Herman Kramer** (TU Delft) and **Jan Meijer** (Akzo Nobel), who are both close to their retirement.

Herman opened the morning session with an overview of 30 years of academic research in the area of industrial crystallization, aiming at a more thorough understanding of processes like transient effects in continuous crystallization, primary and secondary nucleation, crystal growth, monitoring and control of particle size and shape, using process intensification techniques in crystallization. This was followed by a presentation by **Wester de Poel** (Radboud University, Nijmegen) presenting results of using metal-organic framework structures as a host for guest molecules having difficulty to crystallize. Then **Rohit Kacker** (TU Delft) showed results on the application of microwaves during batch crystallization to control nucleation.

Herman Kramer presents.

Jan Meijer talking about salt.

In the afternoon, **Jan Meijer** gave an overview of the last two decades of his 32-year career at Akzo Nobel, Deventer, where we was mainly involved in research on salt, more particularly in the search for a new anti-caking agent that does not interfere with the electrolysis of salt in the production of chlorine. **Adil Acun** (Univ Twente) visualized the real-time 2D growth of hexagonal boron nitride using low energy electron microscopy. **Hao Su** (TU/e) presented a cryogenic TEM study on the crystal growth of nanometers sized crystals of ammonium cobalt kambaldaite, relevant for catalysis. Crystallization processes taking place in concrete, or other porous construction materials, may lead to damage to buildings. **Mohsin Qazi** (UvA) explained how the use of surfactants influences crystallization in pores which lead to confined growth. The last presentation from **Weiwei Li** (TU Delft) discussed the application of a strong, inhomogeneous electric field to enhance the separation of two different crystalline pharmaceutics present in a suspension by selectively collecting these species either on the anode or the cathode.

The program closed with a labtour showing part of the facilities within the section IRS: the oscillatory baffled tube reactor, encapsulation techniques, electric field enhanced separation and microfluidic set-up for determining primary nucleation parameters.

Summarizing, this DACG symposium (once more) showed the diversity in scientific and applied areas of research where crystallization plays an important role and how process intensification techniques can be used to improve or enhance crystallization-related processes. Each of the presentations ended with a lively scientific discussion, which continued during lunch, coffee break and drinks afterwards. Particularly the lectures of Herman and Jan may have helped young scientists to get a feeling of how their career, after obtaining an MSc or PhD degree in physics/chemistry/chemical engineering, could develop in future, either in an academic or industrial environment.

The DACG Spring Meeting was attended by 56 participants, of which 41 from university and 15 from industry. We also welcomed four participants from abroad (Belgium, UK and Germany).

Antoine van der Heijden and Hugo Meekes

Symposium 'Crystallization of Bio- & Pharmaceutical Molecules'

DACG Annual Meeting and Symposium; JNJ, Beerse, Belgium, October 6, 2017

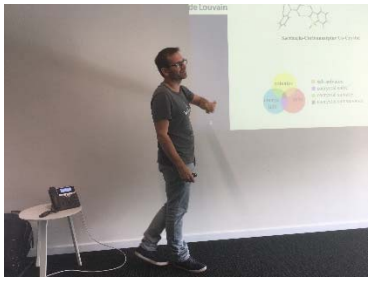
On October 6, 2017, the DACG organized its Annual Meeting and Symposium, hosted by Rob Geertman, JNJ Pharmaceuticals, Beerse, Belgium. The theme of the symposium was "Crystallization of Bio- and Pharmaceutical Molecules", which was prepared in close cooperation with our Belgian colleagues Tom Leyssens (UCL, BE) and Dominique Maes (VUB, BE). Hans te Nijenhuis (president DACG) welcomed the almost 40 participants, mainly from Belgium and The Netherlands. This was followed by a presentation by **Rob Geertman** giving an overview of the company's history, the timeline of the development of new drug molecules from discovery through clinical trials and final drug approval, as well as some of the crystallization challenges when producing drugs. This was followed by a presentation by **Christos Xiouras** (UCL, BE) showing results of applying process intensification techniques (ultrasound and microwaves) in chiral crystallization and deracemization. In the last presentation before lunch, **Mireille Smets** (Radboud University Nijmegen, NL) showed that one of the solid-state polymorphic transitions of pyrazinamide can be inhibited with an additive.

Rob Geertman

Christos Xiouras

Mireille Smets

Directly after lunch the DACG members convened for their DACG Annual Meeting. Other participants had the opportunity to take a guided labtour to the crystallization facilities of JNJ. The symposium continued with a presentation by **Tom Leyssens** (UCL, BE) during which he explained the thermodynamic and kinetic aspects of the application of solution co-crystallization. **Arno Bode** (Aspen Oss, NL) shared his experience on the simultaneous crystallization of an active pharmaceutical ingredient (API) and its stabilizer. As a last speaker in this symposium, **Dominique Maes** (VUB, BE) presented an overview of the research on classical and two-step nucleation of protein crystals. Each of the presentations ended with a lively scientific discussion, which continued during lunch, coffee break and drinks afterwards.



Tom Leyssens

Arno Bode

Dominique Maes

The participants that were not able to join the labtour directly after lunch, were given a second opportunity after the last presentation.

The DACG Symposium was attended by 37 participants, of which 24 from university and 13 from industry. We also welcomed participants from Belgium (11) and France (1). We kindly acknowledge the hospitality of JNJ and especially Rob Geertman for hosting the DACG annual meeting and symposium.

Antoine van der Heijden

Annual Report Dutch Association for Crystal Growth (DACG)
October 2016 – October 2017

Secretariat
Prof dr Antoine van der Heijden
TU Delft
Leeghwaterstraat 39
2628 CB Delft
Tel.: +31 (0) 6 20 43 60 39
E-mail: a.e.d.m.vanderheijden@tudelft.nl
Web: www.dacg.nl

Members
The total number of members is ca. 60.

Board
The DACG Board is summarized in the table below. The Board had five meetings: one face-to-face meeting (hosted by PANalytical, Almelo) and four telcons.

Role	2016 – 2017	E-mail	Appointment deadline
President	Hans te Nijenhuis	hans.te.nijenhuis@panalytical.com	Step down Oct 2018
Secretariat	Antoine van der Heijden	a.e.d.m.vanderheijden@tudelft.nl	Step down Oct 2019
Treasurer	Pieter Vonk	pieter.vonk@dsm.com	Step down Oct 2018
FACET, webmaster	Arie van Houselt	a.vanhouselt@utwente.nl	Step down Oct 2017 [a]
Member	Hugo Meekes	h.meekes@science.ru.nl	Step down Oct 2017 [b]

[a] Not re-electable; call for new candidate(s).
[b] Re-electable for a next period.

The meeting unanimously re-elects Hugo Meekes for a next period of 3 years. Marketta Uusi-Penttilä had aplied as a candidate DACG board member to replace Arie van Houselt. Marketta has an extensive background in crystallization and has experience as a board member in a Swedish educational association. Marketta is elected unanimously by the meeting and Hans te Nijenhuis welcomes her as a new board member for a period of 3 years.

FACET Newsletter
The DACG Newsletter, FACET, was issued in March and December 2017. The objective of the newsletter is to stimulate the communication between scientists and users in the area of crystallization in the Netherlands. The newsletter publishes summaries of relevant PhD theses, upcoming events related to crystallization (conferences, symposia), highlights in crystal growth research and other activities relevant to crystal growers. Several academic scientists have been requested to collect news from their network/colleagues as input to the newsletter, but also members may submit input. Furthermore, initiatives, decisions and plans of the DACG will be published in the FACET Newsletter. FACET is issued electronically or can be downloaded from the DACG website.

Website
The DACG website (www.dacg.nl) provides information regarding the structure and activities of the association. All issues of the FACET Newsletter since 2000 are available electronically; links to Dutch research groups in the area of crystallization are available as well as those of foreign DACG 'sister' associations. We welcome any suggestions for improvements; please contact Hugo Meekes.

Activitities

A shortlist of past activities relevant to the DACG community:

- October 13, 2016, KNCV-event “Avond van de Chemie”, Stadsgehoorzaal Leiden, http://www.kncv.nl/k/news/view/94897/872/save-the-date-13-oktober-avond-van-de-chemie.html
- Gordon Research Conference “Crystal Growth & Assembly”, June 25-30, 2017, Biddeford, USA; http://www.grc.org/programs.aspx?id=12674
- Summer School “Chiral Crystallization, Resolution & Deracemization”, July 3-6, 2017, Nijmegen, NL; http://www.dacg.nl/summerschool_2017.pdf
- 20th International Symposium on Industrial Crystallization, September 3-6, 2017, Dublin, Ireland: http://isic20.com/

Meetings / excursions

On April 7, 2017 the DACG organized a Spring Meeting “Crystallization & Process Intensification”, hosted at the Delft University of Technology. The DACG Fall Meeting “Crystallization of Bio- & Pharmaceutical Molecules” was held on October 6, 2017 and hosted by JNJ, Beerse, Belgium.

KNCV/DACG Crystal Growth Award

In August 2017 the DACG Board submitted a request to the KNCV Board to reintroduce the KNCV/DACG Crystal Growth Award. This request was approved by KNCV. The award, consisting of a certificate and an amount of € 1,000, will be handed out every three years. A committee will be installed that will judge the nominations according to the award requirements. The award is foreseen to be handed out during the DACG Annual Meeting in October 2018.

Strengthening the relationship with KNCV

On March 3, 2017, the KNCV Board organized a special event to strengthen their relationships with the KNCV sections. Also a Meet & Greet was organized 13 June 2017 where Board members of all KNCV sections could meet and get acquainted. KNCV is able to provide support for e.g. section websites and member administration. Furthermore, activities or other news from the KNCV sections can be announced on the KNCV website and/or in C2W.

Upcoming activities

- DACG Spring Meeting, 6 April 2018, AMOLF, Amsterdam
- DACG Fall Meeting, October 2018; location and date to be decided

Membership

Becoming a member of the DACG is easy: send an e-mail message to Antoine van der Heijden.

The annual membership fee is € 15 for professionals, € 10 for retired people and € 5 for (PhD) students. Participating in one of the biannual symposia for the first time automatically includes a membership for one year.

Announcement KNCV Piet Bennema Prize for Crystal Growth

Every three years the Dutch Association for Crystal Growth DACG distinguishes a young researcher with the KNCV Piet Bennema Award for Crystal Growth for his high-level scientific research in the field of crystal growth. The prize is intended for the author of the best dissertation or other scientific publications (or a series thereof) that have been processed in an industrial context and are of similar importance to a dissertation. The prize consists of a certificate and a sum of money of € 1000.

Piet Bennema

Piet Bennema (1932-2016) is one of the founders of the study of crystal growth in the Netherlands. As a professor of chemistry of the solid state from 1976 until his retirement in 1998, he was affiliated with the Radboud University Nijmegen. In this period he elaborated on the theoretical concepts of crystal growth, leading to an improved understanding of the role of the bond strength in the prediction of the morphology of crystals and the role of supersaturation, two-dimensional nucleation, kinetic roughening and spiral growth during crystal growth processes. For his important contributions to the field of crystal growth, he was awarded the Frank Award in 1995 by the International Organization for Crystal Growth.

Candidates

For the award of the prize both young Dutch and non-Dutch are eligible who have largely carried out their research in the scientific field of crystal growth at a Dutch university or a Dutch company.
The candidate must have shown great competence as a researcher as well as scientific originality and productivity. Moreover, it must have a good understanding of the problems of the field.
For candidates who want to be considered on the basis of their dissertation, the dissertation must have been successfully defended at a Dutch university in the three years prior to the closing of the submission deadline. For academic publications, it also applies that these must have been published for the most part in the preceding three-year period.

Nomination

Supervisors can nominate candidates by means of a letter of recommendation with the thesis or other scientific publications, as well as a motivation. The nomination must be submitted in triplicate to the secretary of the DACG. The submission period for the KNCV Piet Bennema Prize for Crystal Growth is from **1 January 2018 to 30 April 2018**.

Award presentation

The KNCV Piet Bennema Award for Crystal Growth is awarded by the KNCV Board on the recommendation of the DACG Board. To this end, the board of the DACG appoints an assessment committee of three experts, who will test the candidates against their competence in the research of crystal growth, scientific quality, productivity and originality and insight into the field.
The presentation of the prize and the associated certificate will take place during the autumn meeting of the DACG in October 2018.

On behalf of the board of the DACG

Prof. dr. A.E.D.M. van der Heijden
Secretary DACG
Delft University of Technology
Dept. Process & Energy
Leeghwaterstraat 39
2628 CB Delft
a.e.d.m.vanderheijden@tudelft.nl

Regulations for the KNCV Piet Bennema Prize for Crystal Growth

1. The Board of the KNCV awards a prize once every three years, consisting of a certificate and a sum of € 1000 to a young researcher for high-level scientific research in the field of crystal growth.

2. The prize is intended for the author of the best dissertation or other scientific publications (or a series thereof) that have been processed in an industrial context and are of similar importance to a thesis. The dissertation must be successfully defended at a Dutch university during the three-year period preceding the selection procedure. The scientific publications should also have appeared in the previous three-year period.

3. The prize will be awarded by the KNCV Board on the basis of a recommendation issued by the Board of the Netherlands Association for Crystal Growth. If the board of the association does not nominate a candidate, no prize will be awarded.

4. The KNCV Board may adopt or reject the recommendation referred to under 3. above. In the latter case the prize is not awarded.

5. Researchers who have carried out their research largely at a Dutch university or at a Dutch company are eligible for the award of the certificate.

6. The requirements for the award are that the candidate must have shown great competence as a researcher as well as scientific originality and productivity. In addition, he / she must have a good understanding of the problems of the field.

7. The Board of the Dutch Association for Crystal Growth ensures that all activities in the context of points 1 to 6 of these regulations are carried out.

8. The Board of the Dutch Association for Crystal Growth will be supported in its work by three experts, from whom it can be expected that they have a good overview of the work of researchers in the field of crystal growth. The experts provide their advice in writing, with documentation, such as publications. Members of the assessment committee cannot nominate candidates themselves.

9. The presentation of the KNCV Piet Bennema Prize for Crystal Growth takes place during the annual meeting of the Dutch Association for Crystal Growth. Justification of the award will be published after the award ceremony in the Chemisch2Weekblad

Recent publications

- Jos J. M. Lenders, Lukmaan A. Bawazer, David C. Green, Harshal R. Zope, Paul H. H. Bomans, Gijsbertus de With, Alexander Kros, Fiona C. Meldrum and Nico A. J. M. Sommerdijk, *Combinatorial Evolution of Biomimetic Magnetite Nanoparticles*, Adv. Funct. Mater., 27 (2017) 1604863
  Affiliations:
  - Laboratory of Materials and Interface Chemistry and Center of Multiscale Electron Microscopy, Department of Chemical Engineering and Chemistry, Eindhoven University of Technology
  - Institute for Complex Molecular Systems, Eindhoven University of Technology
  - School of Chemistry, University of Leeds
  - Department of Supramolecular & Biomaterials Chemistry, Leiden Institute of Chemistry, Leiden University

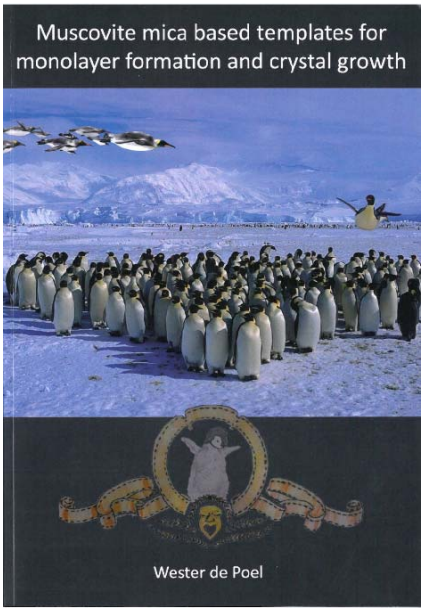
  Inspired by Nature's capacity to synthesize well-defined inorganic nanostructures, such as the magnetite particles produced by magnetotactic bacteria, genetic algorithms are employed to combinatorially optimize the aqueous synthesis of magnetite (Fe_3O_4) nanoparticles through the action of copolypeptide additives. An automated dispensing system is used to prepare and rapidly screen hundreds of mineralization reactions with randomized conditions, varying ferrous iron, base, oxidant, and polypeptide chemistry. Optimization over multiple generations allows identification of conditions under which the copolypeptides promote magnetite formation where this does not occur in their absence. It is found that nanoparticle size, size distribution, and shape can be tuned by the concentrations and compositions of the copolypeptides, and that the reaction pH is the most important factor in controlling the crystalline phase. This approach should be broadly applicable to the syntheses of solid-state materials and represents a valuable strategy for extending biomimetic mineralization to the production of technological materials.

- P. Bampoulis, K. Sotthewes, M.H. Siekman, H.J.W. Zandvliet and B. Poelsema, *Graphene visualizes the ion distribution on air-cleaved mica,* Sci. Reports, 7 (2017) 43451.
  Affiliations:
  - Physics of Interfaces and Nanomaterials, MESA+ Institute for Nanotechnology, University of Twente
  - Physics of Fluids and J.M. Burgers Centre for Fluid Mechanics, MESA+ Institute for Nanotechnology, University of Twente

  The distribution of potassium (K^+) ions on air-cleaved mica is important in many interfacial phenomena such as crystal growth, self-assembly and charge transfer on mica. However, due to experimental limitations to nondestructively probe single ions and ionic domains, their exact lateral organization is yet unknown. We show, by the use of graphene as an ultra-thin protective coating and scanning probe microscopies, that single potassium ions form ordered structures that are covered by an ice layer. The K^+ ions prefer to minimize the number of nearest neighbour K^+ ions by forming row-like structures as well as small domains. This trend is a result of repulsive ionic forces between adjacent ions, weakened due to screening by the surrounding water molecules. Using high resolution conductive atomic force microscopy maps, the local conductance of the graphene is measured, revealing a direct correlation between the K^+ distribution and the structure of the ice layer. Our results shed light on the local distribution of ions on the air-cleaved mica, solving a long-standing enigma. They also provide a detailed understanding of charge transfer from the ionic domains towards graphene.

- Thesis:
  Wester de Poel, *Muscovite mica based templates for monolayer formation and crystal growth*.
  On 23 October 2017 Wester de Poel defended his PhD thesis at the Radboud University in Nijmegen.

  Link: http://repository.ubn.ru.nl/bitstream/handle/2066/176715/176715.pdf?sequence=1



Summary

The goal of the research described in this thesis was to find a suitable substrate for protein crystallization in order to improve crystal size, the number of crystals, crystal quality, and nucleation speed. There are approximately 20.000 different protein molecules in the human body alone, all with a different function. Only a part of these have been crystallized so far. Protein crystals can be used to derive the structure using X-ray diffraction if the crystals are of sufficient size and quality. The structure can be used to derive the function of the protein, and provides insight into the working of the human body at the molecular level. This understanding could be of use in the development of drugs to treat diseases such as Alzheimer.

A substrate can be of use to crystallize proteins that would not crystallize otherwise. Muscovite mica was used as the basis of such a substrate because it is flat over large areas (chapter 2). The first approach to create a network of molecules on muscovite mica to create an ordered template with lattice sizes approaching protein crystal lattice size dimensions was using crown-ethers (chapter 4). These molecules can specifically bind to metal ions on the muscovite mica surface, thereby stabilizing the layer. Unfortunately this molecular layer was not stable under protein crystallization conditions, and therefore unsuitable in view of this application.

The surface of muscovite mica can be functionalized in other ways. The roughness can be tuned by evaporating a molecule onto the surface (chapter 6), and the surface metal ions can be exchanged (chapter 3). Furthermore, the chemical functionality can be tuned by applying a thin molecular layer of organothiols (chapter 5), and the periodicity can be tuned by applying a thin polymer layer (chapter 9). These thin layers were investigated using surface sensitive techniques (AFM, SXRD, and XPS). The influence of functionalization on the crystallization behavior of proteins was investigated (chapters 6-9).

The three investigated parameters (roughness, chemical functionality, and periodicity) also provided

many examples where a change in the investigated parameter did not significantly change the crystallization outcome. The proteins talin and albumin crystallized equally well on surfaces with varying roughness, the chemical functionality of a surface did not influence the crystallization of talin, and in many cases zeolites did also not affect the protein crystallization outcome.

There were a few diversions during this research period, and in two cases this has led to a chapter. The growth of gallium nitride on muscovite mica was investigated (chapter 11). Papers reported the ordered (epitaxial) crystal growth of this material on muscovite mica. If a method could be found to grow large epitaxial crystals on muscovite mica, this would probably lead to applications, as gallium nitride is used in many devices. Unfortunately no optimal growth conditions were found, further study is required.

A different diversion led to chapter 10, which is about the reaction of noble metals with organothiol molecules. Originally, thiol molecules were intended to functionalize noble metal surfaces to study the effect of these layers on protein crystallization. When the noble metal surface reacted with the thiols, this was investigated further and resulted in a chapter 10.

The summary given above leads to the conclusion that most likely no universal template for protein crystallization exists. Therefore, in the attempt to crystallize a new protein molecule, surfaces with varying roughness, chemical functionality, symmetry, and periodicity should be used.