

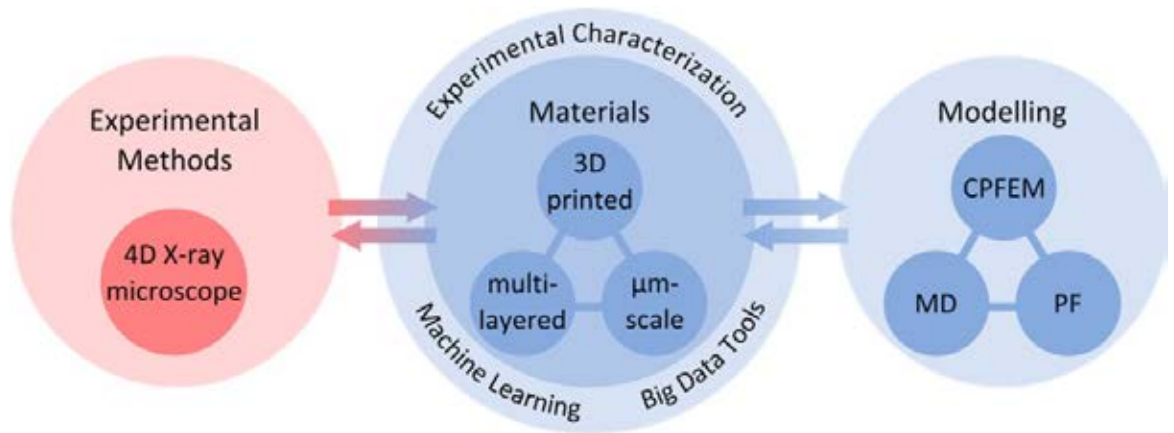
Annual report 2019

M4D
Metal Microstructures
in Four Dimensions



Metal Microstructures in Four Dimensions

The key elements of the project. The **materials research** related elements are shown in blue and **technique development** is shown in red.



The goals are:

- 1) to develop a universal *laboratory-based 4D* X-ray microscope with potentials in the broad field of materials science and beyond;
- 2) to advance metal research by quantifying *local microstructural variations* using the new 4D tool and by including the effects hereof in the understanding and modelling of industrially relevant metals.

Today, high resolution 4D (x,y,z,time) crystallographic characterization of materials is possible only at large international facilities. This is a serious limitation preventing the common use. The new technique will allow such 4D characterization to be carried out at home laboratories, thereby wide spreading this powerful tool.

Whereas current metal research mainly focuses on average properties, local microstructural variations are present in all metals on several length scales, and are often of critical importance for the properties and performance of the metal. In this

project, the new technique will be the cornerstone in studies of such variations in three types of metallic materials: *3D printed, multilayered and micrometre-scale metals*. Effects of local variations on the subsequent microstructural evolution will be followed during deformation and annealing, i.e. during processes typical for manufacturing, and occurring during in-service operation.

Current models largely ignore the presence of local microstructural variations and lack predictive power. Based on the new experimental data, three models operating on different length scales will be improved and combined, namely *crystal plasticity finite element, phase field and molecular dynamics models*. The main novelty here relates to the full 4D validation of the models, which has not been possible hitherto because of lack of sufficient experimental data.

The resulting fundamental understanding of the inherent microstructural variations and the new models are foreseen to be an integral part of the future design of metallic materials for high performance applications.

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Introduction



We just received the happy message that our ERC Advanced Grant application was approved.

This introduction covers both 2018 and 2019 since we did not make an annual report in 2018.

The years 2018 and 2019 will for us always stand out as the years we were awarded an ERC Advanced Grant called Metal Microstructures in 4D, and were very well received in our new home Department: DTU Mechanical Engineering. Both have turned out to be excellent for us.

The two years have shown that an ERC Advanced Grant is connected with extraordinary attention and recognition. The Grant is large enough to build an outstanding research group. At present, we are six senior and four junior scientists plus three technical/administrative staffs working full or part time on the project. In addition to that, one PhD student (funded by FTP) and seven long term visiting PhDs and postdocs are part of our team. And the group is growing, which is optimal considering the research plans and ideas we have.

The three overarching objectives of the M4D ERC project are:

- i. To develop a versatile 4D (x,y,z , time) X-ray microscope with a spatial resolution 10 times better than is currently possible on *laboratory-based* systems.

- ii. To explore, analyze and quantify *local* microstructural variation on appropriate length scales and incorporate these variations in the understanding of the subsequent microstructural evolution during plastic deformation and annealing of metals.
- iii. To develop a series of much improved models for plastic deformation and annealing of metals by full 4D validation, and to establish methodologies to couple the models into a united multi-scale tool.

The M4D center activities started in October 2018, and since then we have had good progress related to all three objectives. Some of the highlights include:

- By using a simple geometrical magnification possibility, our simulations suggest a 3.5 times improvement in resolution of laboratory diffraction contrast tomography (LabDCT™).
- We have characterized 3D printed maraging steel samples and shown that the heat treatments needed for property optimization are different in printed and in conventionally manufactured samples.
- Concerning the third objective, the M4D home team at DTU are not experts in advanced simulations or modelling. Therefore, three foreign experts in crystal plasticity finite element modelling, phase field and molecular dynamics simulations are affiliated partners in the M4D center. A highlight of the year was a two-day joint meeting with these experts. The meeting was focused on how exactly we could best couple our experimental and their simulation work. The two days were very intense, highly interesting and the outcome was a series of prioritized joined subprojects, which we consider essential to reach the third objective above. The first results on phase field simulations of recrystallization boundary migration through a theoretical, yet realistic, inhomogeneous deformation microstructure are now already almost ready for publication.

For further information on these and more scientific highlights, please read the following pages.

Another major highlight of the year was the Risø International Symposium on Materials Science, which we organized. It was the 40th in this series of Symposia and held during the first week of September 2019. The theme of the Symposium was “Metal Microstructures in 2D, 3D and 4D” and was planned as the international kick-off of our M4D center activities. We all participated in the Symposium and found it to be very stimulating, inspiring and fruitful. It gave us new ideas, fostered more collaborations and further documented that our M4D ideas, results and plans are of international interest.

In August, four of us participated in the 7th International conference on Recrystallization and Grain Growth, which was held in Gent, Belgium with 270 participants. We enjoyed the conference and were happy to present our work in three invited and one contributed talks dealing with inhomogeneous deformation, recovery, recrystallization in 4D and the surprising fact that also recrystallizing grains may contain residual stress.

During the two years, our collaboration with China, in particular Chongqing University and Tsinghua University, has been further strengthened and formalized by a 1,000 Talent-Foreign Expert Grant and another similar contract with Chongqing University (CQU). Besides extended stays for the Danish scientists in China, this has significantly increased the number of long term guest PhD students from CQU working in the M4D center. CQU hosts one of the newest and most advanced electron microscope centers in the world, and the scientists at CQU focus on several research topics related to our work. The collaboration is thus of significant mutual benefit.

At the start of 2018, we changed our affiliation from DTU Wind Energy on Risø Campus to DTU Mechanical Engineering on Lyngby Campus. We are happy about this change. We can now look for much wider applications of our research, and we have gotten new colleagues who are experts in, for example, manufacturing; an essential supplement to our knowledge, which is mostly within materials characterization and analysis. Almost all the experimental equipment we are responsible for including electron microscopes and metal processing equipment, is still at Risø. To allow efficient use of this equipment, we have offices in both locations. In Lyngby we are in building 425 and at Risø we fill the entire so-called Microscopy house - i.e. guest students, junior and senior staffs have offices next to each other. Altogether, this is a nice and effi-

cient solution and we are happy being part of the Department of Mechanical Engineering.

Since joining our new department, we have managed to secure significant additional funding - see the inserted box. This funding supports research, which supplements the ERC M4D project and substantial synergy is foreseen. Furthermore, several of these additional projects are more application oriented than the ERC one.

We therefore look very positively towards the future and are thankful that the funding from the ERC grant and the welcoming to DTU Mechanical Engineering have allowed us to take a significant step forward in science and its application.

External Funding granted during the years 2018 and 2019

M4D: Metal Microstructures in Four Dimensions, European research Council Advanced Grant, 2.496.519 Euro, 2018-2023, PI: Dorte Juul Jensen

DMDS: Demonstration of Modular Drive System, Energiteknologisk Udviklings- og Demonstrationsprogram (EUDP), 1.769.568 DKK (DTU part), 2019-2021, PI's at DTU: Dorte Juul Jensen + Niels Leergaard Pedersen

rs3DXRM: Developing a laboratory X-ray technique for local residual stress measurements and design of advanced metallic materials, DFF FTP1, 2.453.835 DKK, 2018-2021, PI: Yubin Zhang

Nippon Steel: Collaborative research on quantitative description of microstructures and strain distribution in steel materials with advanced analysis techniques, Industrial Contract with Nippon Steel, 1.500.000 DKK, PI: Dorte Juul Jensen

REPOWDER: Design of metal powders for 3D printing - from scrap to resource, DFF FTP2, 4.944.694 DKK, PI: Dorte Juul Jensen

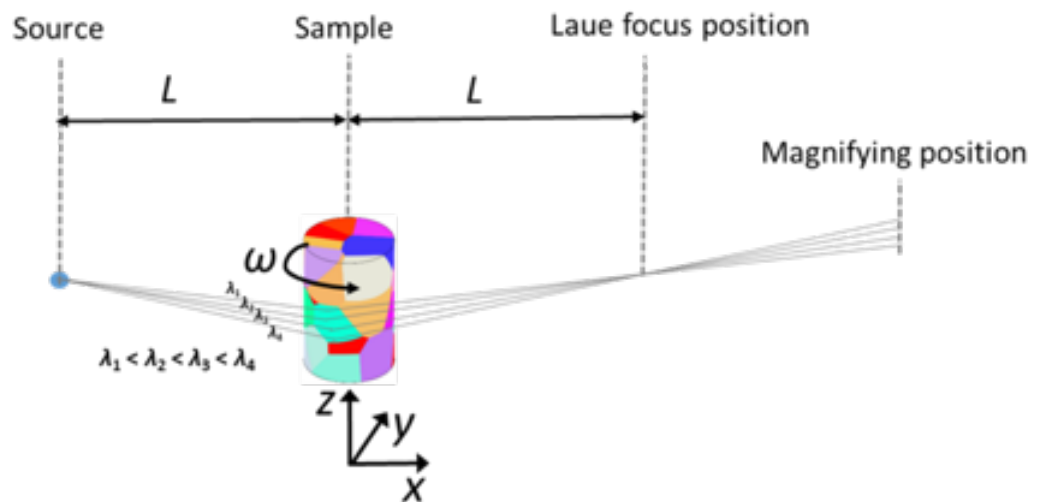
LapXRD: Bringing synchrotron micro-diffraction to the laboratory, Villum Experiment, 1.944.811 DKK, PI: Yubin Zhang

Exlime: Explore the strength limit of metals, Villum Experiment, 1.998.828 DKK, PI: Xiaodan Zhang

Highlight

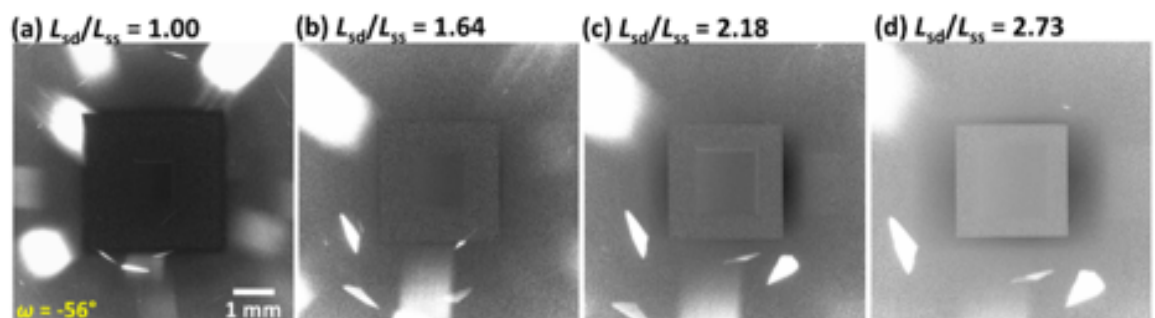
Improvement of the spatial resolution of laboratory X-ray diffraction contrast tomography

A sketch of LabDCT showing the principle of Laue focusing and of the suggested geometrical magnification. An X-ray beam with wavelengths between λ_1 and λ_4 focus into a line at the Laue focus position. The diffraction pattern can be magnified by increasing the sample-to-detector distance. Hereby, the spatial resolution can be improved by a factor of three or more.



Laboratory diffraction contrast tomography (LabDCT) has recently enabled non-destructive 3D characterization of grain orientations, sizes and shapes in bulk materials. LabDCT uses X-rays generated from a conventional X-ray tube to illuminate the sample. Such a beam is divergent and polychromatic. A grain in the sample acts like cylindrical lens and focuses the X-rays into a line on the detector when sample-to-detector distance (L_{sd}) equals to sample-to-source distance (L_{ss}). This is the so-called Laue focusing effect and the conventional LabDCT measurements are performed in this condition. This limits the spatial resolution of the LabDCT.

To make LabDCT a more versatile tool, we have investigated possibilities to improve the spatial resolution by increasing the ratio between L_{sd} and L_{ss} to magnify the diffraction spots. It is shown that in a partially recrystallized Al sample the sizes of diffraction spots can be magnified by factors between 2 and 3.5 when $1.64 \leq L_{sd}/L_{ss} \leq 2.18$. This ensures a good balance between obtaining a considerable geometrical magnification and a sufficient number of diffraction spots. The resulting grain reconstructions show that the increase of L_{sd}/L_{ss} to magnify the diffraction spots has great potentials to improve the spatial resolution of LabDCT.



LabDCT projections showing the geometrical magnification of diffraction spots as a function of L_{sd}/L_{ss} .

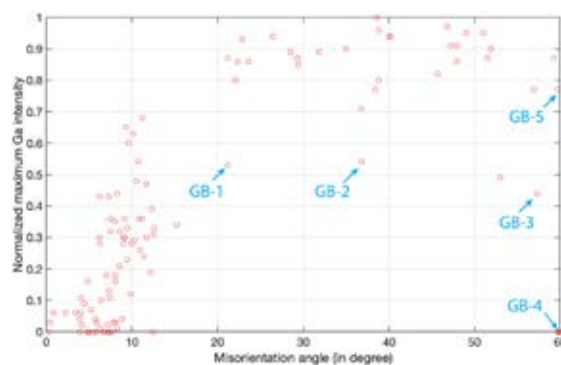
Highlight

Grain boundary wetting studied by multimodal X-ray tomography

By the commercially available ZEISS Xradia 520 Versa X-ray microscope equipped with the laboratory diffraction contrast tomography (LabDCT™) module in the DTU CASMAT center, it is possible to map non-destructively in 3D *both* the grain structure including the crystal orientations and features with a different X-ray absorption contrast than the rest of the sample, for example second phases or particles.

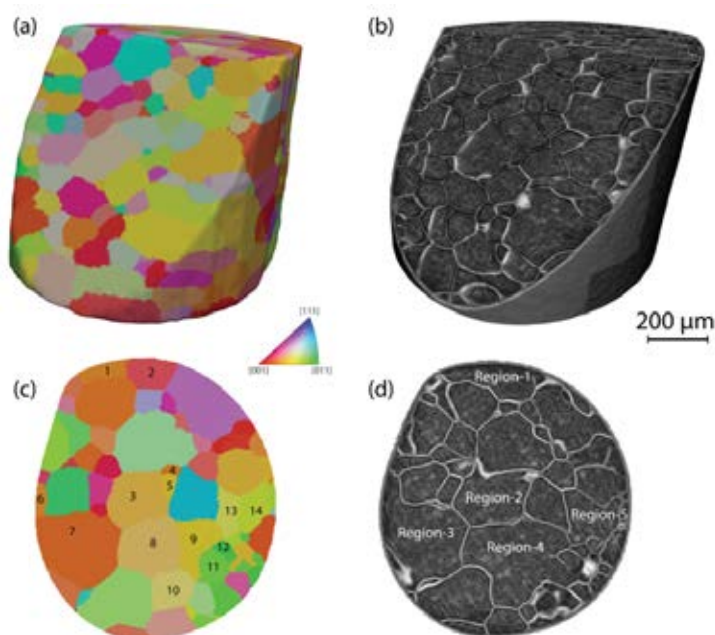
In the present work, we used this multimodal opportunity to correlate grain boundary wetting with the 3D characteristics of the boundaries in Al (AA 1050) exposed to liquid Ga. The grain boundary structure was first characterized by LabDCT and subsequently, after the Ga had penetrated the sample, absorption contrast tomography (ACT) was used to map the distribution of Ga. The grain boundary plane and misorientation determined based on LabDCT data for 115 boundaries were

correlated to the normalized Ga intensity in the ACT scans. It was found that boundaries with similar misorientations can be very differently wetted and wetting is therefore not determined by the boundary misorientation only (as it is often assumed). Instead, it is suggested that it is the grain boundary energy, which determines the preferential Ga penetration path in the Al matrix.



Normalized maximum intensity of Ga plotted as a function of misorientation angle of the grain boundaries. Five grain boundaries (GB-1, GB-2, GB-3, GB-4 and GB-5) with misorientations close to that of ideal CLS boundaries are highlighted.

LabDCT and ACT of the same gauge volume. (a) 3D grain map reconstructed from LabDCT colored according to the crystallographic orientation along the rotation axis of the sample. (b) 3D map of the Ga distribution reconstructed from ACT, bright lines reveal the Ga-decorated grain boundaries. 2D images of a sample section perpendicular to the sample rotation axis reconstructed from LabDCT (c) and ACT (d). Regions 1–5 marked in (d) are regions with some of the grain boundaries not penetrated by Ga. The non-wetted grain boundaries are revealed from the LabDCT map in (c).



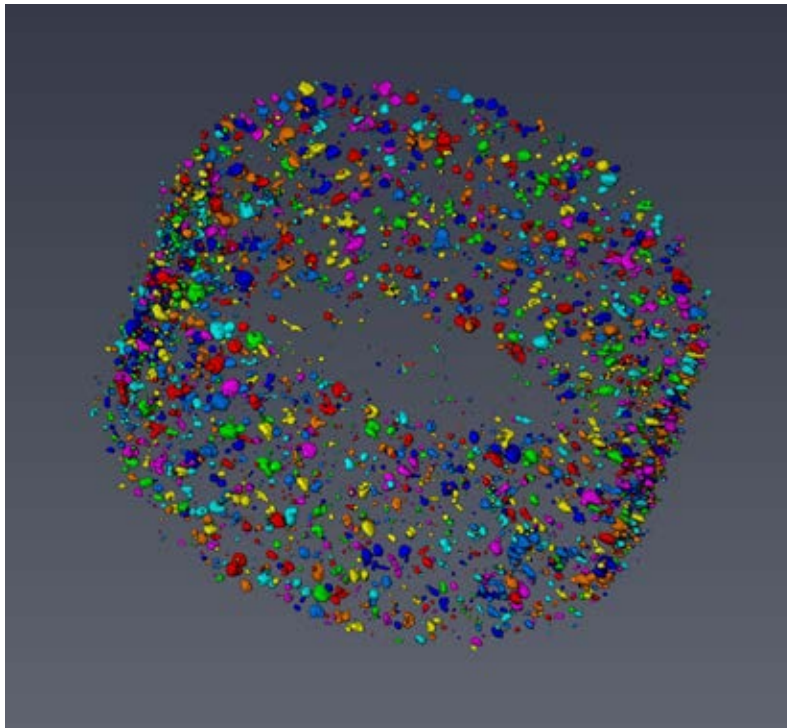
Highlight

Microstructures and properties of 3D printed maraging steel

Additive manufacturing (AM), also known as 3D printing, is a new popular manufacturing process, currently undergoing rapid growth. The new process provides many possibilities for new designs in manufacturing together with new challenges, for example, there are small voids in 3D printed metals (see figure below).

One of the materials often used in 3D printing is maraging steel. This is a popular material because of its good weldability and superior mechanical properties, which make it ideal for tool manufacturing. Our work shows that the 3D printing has a large effect on the microstructural evolution and

on the mechanical properties of maraging steel. Heating of the 3D-printed samples at a moderate temperature leads to precipitation and thus strengthening - a phenomenon similar to that in conventionally manufactured samples. However heating at a moderate temperature leads to concurrent reversion of austenite in 3D-printed samples but not in conventionally manufactured samples, and therefore different heat treatment is needed for property optimization of maraging steel depending on the manufacturing history. Part of the work was carried out in collaboration with Grundfos A/S through a Master project, where the student was awarded the top mark.



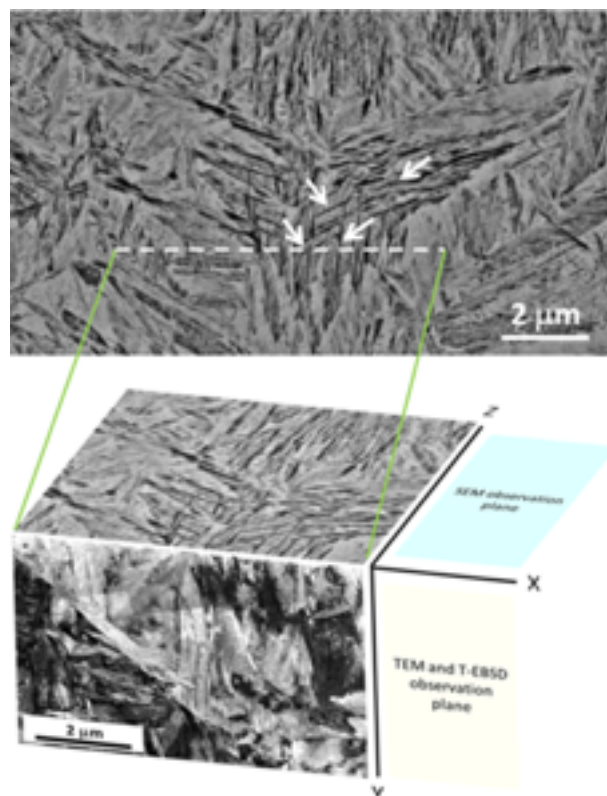
The distribution of voids, shown in random colors, inside a 3D-printed cylindrical sample (2 mm in diameter) of maraging steel characterized by X-ray tomography.

Highlight

Localized deformation in nano-sized martensite associated with micropitting in case carburized gears

Micropitting has long been recognized as the prevailing failure in rolling and sliding contact elements such as gears, roller bearings and camshafts. In collaboration with Dinesh Mallipeddi, Mats Norell and Lars Nyborg, Chalmers University of Technology, Sweden, we followed systematically the evolution of micropitting and its relation to microstructural changes in 12 gear pairs belonging to the same batch tested from 200 to 2.6×10^7 cycles. The aim was to unveil the microstructural reason of micropitting. Through detailed 2D and 3D microstructural characterization

as demonstrated in the figures, localized deformation of nano-sized martensite laths (examples marked by white arrows) without preferential orientations to the gear surface were found to account for the evolution of micro-cracks, leading to micropitting. The deformed martensite laths/plates are seen in bundles and have a thickness below 100 nm, with the majority being less than $2 \mu\text{m}$ in length. This finding sheds new light on the microstructural reason of micropitting by localized deformation of martensite.

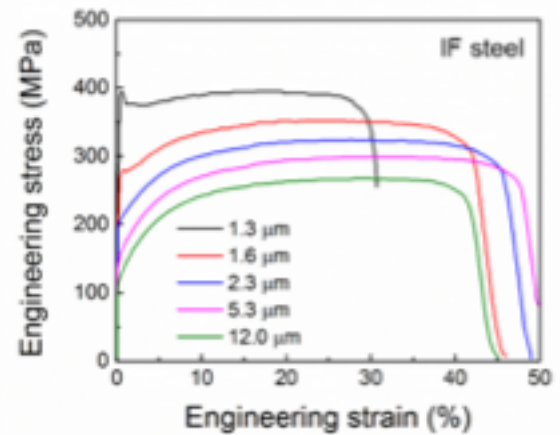
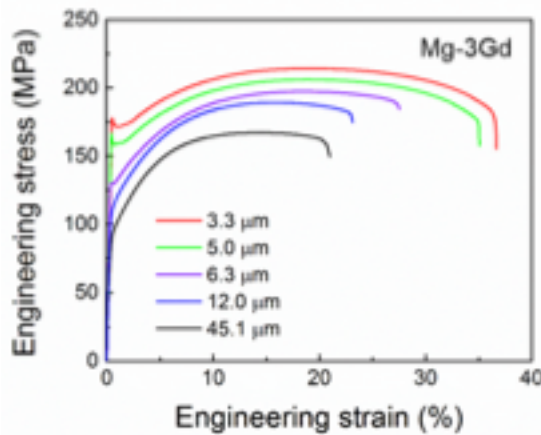


The 2D and 3D characterization of deformation lamellae. The bright field TEM micrograph is from a sample cut parallel to and about $5 \mu\text{m}$ below the tooth surface. The white dashed line represents the middle of the lift-out of the thin foil, with the thickness of around 100 nm, and along the coordinate axis X, at which the SEM observation plane and the TEM/T-EBSD plane intersect with one another.

Highlight

Simultaneous increases of strength and ductility in hcp Mg-3Gd alloy through grain refinement

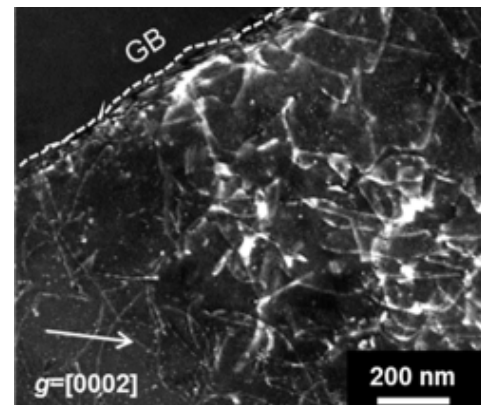
Tensile stress-strain curves of an hcp Mg-3Gd alloy and a bcc interstitial free (IF) steel, showing opposite grain size dependence of strength and ductility: simultaneous increase of both strength and ductility in the former while the so-called trade-off dilemma is observed in the latter.



Magnesium alloys of the hcp crystal structure have many possible slip systems (basal $\langle a \rangle$, prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$) and twinning systems. However, magnesium alloys often show low strength, which is due to the easy activity of basal $\langle a \rangle$ slip given by its much lower critical resolved shear stress (CRSS) than the other systems, and poor ductility, which is due to the limited availability of independent basal slip systems. Alloy design by addition of rare earth elements like Gd can efficiently reduce the difference in the CRSS between basal $\langle a \rangle$ slip and prismatic or pyramidal slip, thus creating possibilities of activating different slip systems. Microstructural design by grain size refinement can also alter the balance between dislocation slip and deformation twinning. A combined use of these two strategies is a novel approach to improve the mechanical behavior of Mg alloys.

Tensile tests of a series of Mg-3Gd alloy samples have shown a strong Hall-Petch strengthening and simultaneous increases of both strength and ductility with decreasing grain size. Deformation mechanism analysis by transmission electron microscopy and electron backscatter diffraction have revealed

that refining the grain size to values below $5 \mu\text{m}$ leads to a drastic transition in the deformation mechanism from basal $\langle a \rangle$ slip and twinning to basal $\langle a \rangle$ and $\langle c+a \rangle$ slip. Enhanced work hardening has been observed in the samples of finer grain sizes because of the strong interactions between dislocations of different Burgers vectors. Activating different deformation mechanisms is considered to be a general strategy to overcome the longstanding strength-ductility trade-off of metals.



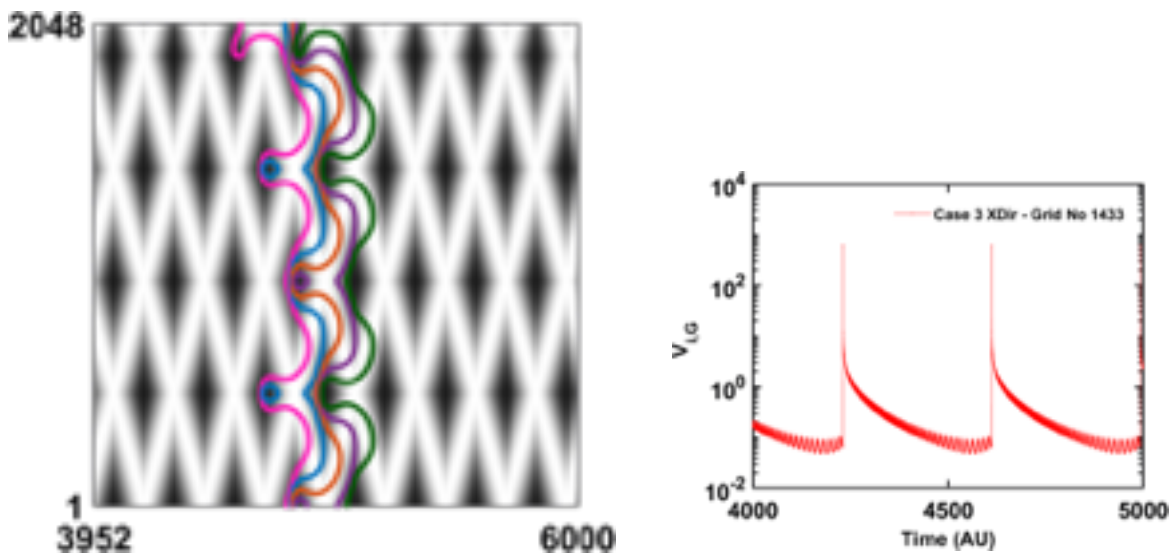
A transmission electron microscopy dark field image showing a high density of $\langle c+a \rangle$ dislocations in a tensile deformed Mg-3Gd sample with a fine grain size of $3.3 \mu\text{m}$. The imaging condition is $g = [0002]$ at which all possible $\langle c+a \rangle$ dislocations are visible while the $\langle a \rangle$ dislocations are invisible.

Highlight

Simulation of boundary migration during recrystallization

In a collaboration with Nele Moelans KU Leuven Belgium, we are using a phase field model to simulate the migration of recrystallization boundaries into two-dimensional varying deformation energy fields. These fields are chosen as idealized versions of typical low to medium strain deformation microstructures with two sets of deformation induced geometrically necessary boundaries. The driving force is thus heterogeneous along the length of the recrystallization boundary and varies as the boundary migrates. The simulations show that

the shape and size of the minimum deformation energy in a given deformation field as well as the energy gradient strongly affect the migration of the recrystallization boundary. Very rough boundary shapes and abrupt changes in the local migration rates are observed. Examples are shown in the figures. The work is part of our long-term aim to obtain an understanding of how the local variations in deformation microstructures affect recrystallization boundary migration. Such insight is difficult to obtain from experiments alone.



Recrystallization boundary migration through an inhomogeneous deformation energy field. Figure (a) shows the positions after a series of 10000 time-steps of an originally flat boundary moving from the left to right. Black and white areas in the deformation field show areas of minimum and maximum energy, respectively. Figure (b) shows the instantaneous velocity of one point of the recrystallization boundary shown in figure (a). The abrupt velocity changes are obvious.



40th Risø International Symposium on Materials Science, 1-6 September 2019.

Public Outreach and Collaboration

The 40th Risø International Symposium

A highlight event was the 40th Risø International Symposium on Materials Science: “Metal Microstructures in 2D, 3D and 4D”, held during the period 2-6 September 2019 at DTU Risø Campus with 80 participants from 13 countries. To be coherent with the core activities of the ERC M4D center, the symposium was planned to address:

- The latest and unprecedented development of 2D, 3D and 4D characterization techniques operating on multiple length scales, down to the atomic dimension and
- Frontier research in the areas of deformation, recovery and recrystallization of metals and alloys fueled by the novel advances in experimental characterization and modelling.

As something new, these topics were addressed by three categories of presentations: keynote, invited and contributed. Traditionally

we have only had two levels. The new model was found to work very well. There were in total 15 keynote lectures including one from the M4D center, 17 invited ones, three of which were co-authored by M4D center staff and 38 contributed presentations. Following the tradition, each presentation is accompanied by a paper published by Institute of Physics-conference series as open access and presented in a Proceedings book of 580 pages given to all participants upon arrival to the Symposium.

Because of the great interest in the Symposium and therefore the high number of presentations, we this year had to have a poster session. We decided to use a format where the posters were on show in the coffee-break room during the entire Symposium week and with snap one minute presentations in plenum to stimulate interest in the individual posters. This format was found to be excellent and stimulating for discussions.

The Symposium gave ample opportunities for the senior and junior members of the M4D center to present ongoing work and discuss it with the other Symposium participants among whom many are experts also in closely related areas. Altogether, the Symposium documented that the theme Metal Microstructures in 2D, 3D and 4D is vivid, vibrant and developing fast, and we are happy to be right in the middle of it.

ERC Kick-Off Meeting

A central part of the ERC M4D project is the tight link between the core experimental work and the related simulations. As we, in the home M4D team, focus on experimental characterization and analysis, we have in the M4D project joined forces with three young, internationally recognized simulation experts. These are Senior Scientist Romain Quey from Ecoles des Mines, Saint Etienne, France on crystal plasticity finite element modelling, Professor Nele Moelans from KU Leuven, Belgium on phase field simulations and Associate Professor Eric Homer from Brigham Young University, USA on molecular dynamics modelling.

To get the collaboration with these affiliated partners off on a flying start, we all met for a two-day kick-off event in Brussels, January 16th-18th 2019. At the meeting, we each presented on-going work of relevance for the M4D project and discussed in great detail what we found to be the most promising ideas for the future collaboration. We agreed upon specific sub-projects to focus on and planned how to exchange the data sets which in most cases are huge.

Besides interest, a successful collaboration always requires sufficient manpower and time spend together. We therefore already at the kick-off meeting decided to share a 1 year Post Doc student between DTU and KU Leuven, half year each place, to send the M4D PhD students to the relevant affiliated partners for 3-6 months and have extended exchange visits for the senior scientists. This plan is successful and already now, four joint papers are in the pipeline.



Kick-off meeting with the affiliated partners.



Staff

Project Leader



Dorte Juul Jensen

Project Administrator



Lone Groes Hede

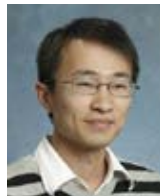
Scientists



Chuanshi Hong



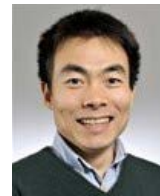
Xiaoxu Huang



Tianbo Yu

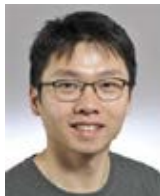


Yubin Zhang



Xiaodan Zhang

Post Docs



Haixing Fang



Jun Sun
until Feb. 2019



Vishal Kapildeo
Yadav

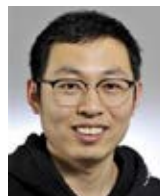
PhDs



Adam Alexander
Lindkvist



Tianzhao Wang
until June 2019



Chunlei Zhang

Technical staff



Gitte Christiansen



Elias Meskouk
Mathiesen

Long term guests (more than a month)

Guest scientists and Post Docs

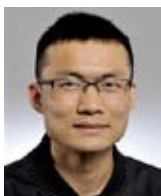


Hongjiang Pan
(Post Doc) from Kunming
University of Science
and Technology, China
Nov. 2019 - Oct. 2021



Yuhui Wang
(Guest Scientist)
from Yanshan
University, China
Feb. 2019 - May 2019

PhDs



Faping Hu
from Chongqing
University, China
Sep. 2019 - Aug. 2020



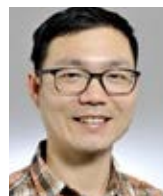
Xiuchuan Lei
Xiuchuan Lei
from Chongqing
University, China
Aug. 2019 - Aug. 2020



Danyang Li
from Harbin Institute of
Technology, China
Aug. 2019 - Feb. 2021



Xuan Luo
from Chongqing
University, China
Mar. 2018 - Mar. 2019



Guoqiang Ma
from Chongqing
University, China
Mar. 2019 - Jun. 2020



Linfei Shuai
from Chongqing
University, China
Mar. 2019 - Jun. 2020



Shuhei Yoshida
from Kyoto
University, Japan
Nov. 2018 - Mar. 2019



Guangni Zhou
from Xian Jiaotong
University, China
Sept. 2018 - Sept. 2019



Wenxing Zhu
from Xi'an Jiaotong
University, China
Nov. 2019 - Nov. 2021

Short term guests (less than a month)

Prof. Andy Godfrey from Tsinghua University, Beijing, China

Mr. Hiroshi Kaido, from Nippon Steel, Tokyo, Japan

Prof. Masakazu Kobayashi from Toyohashi University of Technology, Japan

Mr. Yuji Kubo, from Nippon Steel, Tokyo, Japan

Prof. Qing Liu from JITRI, Nanjing, China

Dr. Vahid Samaeeaghmiyoni from Antwerp University, Holland

Prof. Robert Sanders from Chongqing University, China / Novelis Inc, USA

Dr. Naoyuki Sano from Nippon Steel, Tokyo, Japan

Dr. Anthony Seret from Ecole nationale superieure des Mines de Saint-Etienne, France

M4D publications

- [1] J. Ahlström and X. Zhang, "Effect of strain gradient on the microstructure and mechanical properties of pearlitic steel" in *IOP Conference Series: Materials Science and Engineering*, Volume 580 and *Proceedings of the 40th Risø International Symposium on Materials Science: Metal Microstructures in 2D, 3D and 4D*, 2019, vol. 40, pp. 343-348. (<https://doi.org/10.1088/1757-899x/580/1/012027>)
- [2] N. Chen, G. Ma, W. Zhu, A. W. Godfrey, Z. Shen, G. Wu and X. Huang, "Enhancement of an additive-manufactured austenitic stainless steel by post-manufacture heat-treatment" in *Materials Science and Engineering*, 2019, Volume 759, pp. 65-69. (<https://doi.org/10.1016/j.msea.2019.04.111>)
- [3] H. Fang, D. Juul Jensen and Y. Zhang, "An efficient method to improve the spatial resolution of laboratory X-ray diffraction contrast tomography" in *IOP Conference Series: Materials Science and Engineering*, Volume 580 and *Proceedings of the 40th Risø International Symposium on Materials Science: Metal Microstructures in 2D, 3D and 4D*, 2019, vol. 40, pp. 367-372. (<https://doi.org/10.1088/1757-899x/580/1/012030>)
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