Down to the **atom**

Professor Randi Holmestad and her team are working with aluminium industry insiders to develop solutions for creating and streamlining customised aluminium products by researching the effects of recycling at an atomic level

Can you provide an insight into your research methodology and experimental equipment?

The transmission electron microscope (TEM) is a very well-suited tool for studying materials at the nanoscale. We use electrons to 'see' the atoms in our alloys and, dependent on the electron scattering power of each atomic column, we can image the projected columns in the sample down to sub-nanometre resolution.

We often use a technique called high angle annular dark field scanning transmission electron microscopy (HAADF-STEM), which produces a projected image of the atomic columns in our sample and tells us what types of atoms are in each. In this way, we can see where the added elements preferentially reside and then perform corresponding theoretical calculations and simulations.

Can you explain HAADF-STEM in a little more detail?

In the HAADF-STEM technique, we scan the electron beam across the sample and collect the electrons scattered to high angles. If the sample is well aligned and the probe is small, we obtain high-resolution images showing the columns in the precipitates. Bright columns contain atoms which have a higher atomic number than darker columns due to more high-angle scattering from the heavier atoms. By studying these images, we can explore the precipitates' structure and determine their composition. Such resolution has only been made possible with new aberration-corrected TEMs.

How are ordered and disordered precipitates in Al-Mg-Si-Cu differentiated and how is this contextualised within your overarching goals?

We measure a hardness increase of the Al-Mg-Si alloy when small amounts of Cu are added, and when we look at the resultant microstructure in the TEM we understand why. This is because we find many more and much smaller precipitates in the Cu-added alloy compared to Cu-free ones. When we start to look at the details using HAADF-STEM, we see that the precipitates with Cu are disordered, ie. we do not see a repeated structure in the projection as we do in the Cu-free precipitates. The disordered precipitates give the alloy greater strength.

What conclusions can you infer from the findings of your study?

Our general conclusion is that strength is highly dependent on the type and size of precipitates, and that these can be manipulated with alloy composition and heat treatment. By adding small amounts of one or several elements (such as Cu, Zn, Ag or Ge), we see that strength increases and precipitates form a projected hexagonal silicon network. All precipitates also contain fragments of other precipitate structures and we recognise a few building blocks with the same local geometry.

Through the TEM Gemini Centre, your group at the Norwegian University of Science and Technology (NTNU) is collaborating with SINTEF and an industry partner. Has this partnership proved successful?

The TEM Gemini Centre is a collaboration between the transmission electron microscopy group in the Department of Physics and the Trondheim Materials Physics group in SINTEF. Adding the Norwegian aluminium company Norsk Hydro, we make a nice triangle with different roles. SINTEF and Hydro are more applied and perform confidential research, whereas NTNU performs the open elements of research and publishes findings. In addition, the University provides students and often manages infrastructure and equipment, while SINTEF maintains continuity in competence, with their researchers helping to supervise students and assisting NTNU professors with additional projects.

What challenges have you encountered over the course of your study and have these obstacles provided you with new research avenues?

State-of-the-art infrastructure and equipment have been imperative. For years, we have tried to finance the purchase of new TEM instruments, but it was only when we joined a larger national project group and became a 'national large-scale infrastructure project' that we had momentum and managed to garner support for the purchase of top instruments, in addition to financing rebuilding and other lower level instruments.

The so-called 'Valley of Death' refers to the important transition between research and successful innovation. How is the Centre working to accelerate the development of applied research into commercial enterprise?

I think the close contact we have with industry through industry-initiated projects helps us to bridge this Valley of Death. It can sometimes feel like a long way from atoms to commercial products, but I believe we have managed to join forces well between university, institute and industry – this is what has made our projects successful.

Alloys by design

Physicists at the **Norwegian University of Science and Technology** have been conducting extensive research on precipitate phases in aluminium alloys in a bid to better comprehend the fundamentals responsible for nucleation and precipitation

EVERY YEAR NORWAY alone produces over 1 million tonnes of aluminium alloys. These alloys are a key group of materials with vital properties – including high strength-to-weight ratios, advantageous formability and weldability, and corrosion resistance – making them extremely desirable in the building and construction, automotive, and marine biology industries.

Processing certain aluminium alloys through a heat treatment or precipitation hardening step gives them their strength, as metastable precipitates – small particles of other elements – are developed inside the host aluminium metal during heat treatment. The particles are formed as needles or plates along certain directions. "You can compare the precipitates to steel reinforcements in concrete, but at a totally different scale," suggests Professor Randi Holmestad from the Norwegian University of Science and Technology (NTNU). "The particles we talk about are 10,000 times smaller than a human hair!"

PRECIPITATION AND ALLOYS

The form, structure and strengthening properties of age-hardening precipitates depend on the composition and thermomechanical history of the alloy. Understanding the atomic structure of these precipitates and the impact they have on each other and the physical properties of the material can be used to customise materials to preferred property forms.

A team at NTNU, led by Holmestad, collaborates with SINTEF – the largest independent research organisation in Scandinavia – and Norsk Hydro – a global aluminium company. This collaboration has charted and verified most precipitate phases; their mapping and calculations have been performed by combining advanced transmission electron microscopy (TEM) techniques with atom probe tomography (APT) and computations based on density functional theory (DFT). Holmestad and her colleagues are currently focused on diffusion and solute clustering, nucleation, growth and transformation of precipitates to better grasp their behaviour.

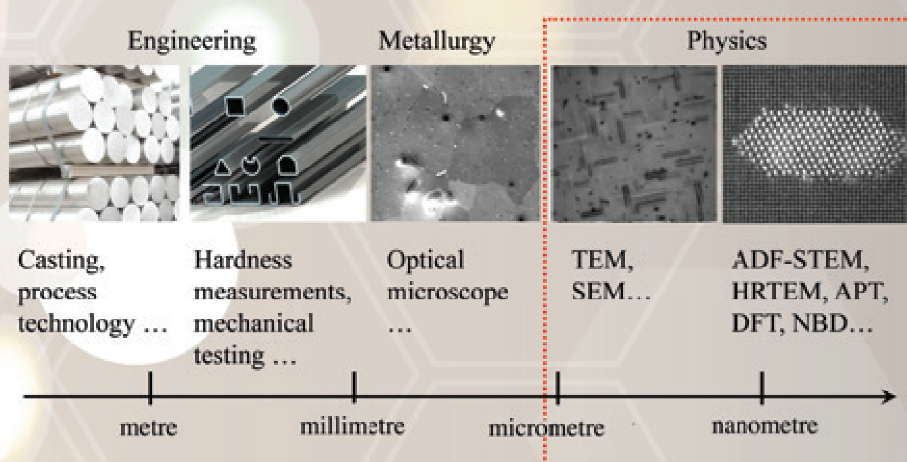
THE PERFECT MICROSCOPE

To fully investigate aluminium precipitates, it is necessary to observe them at their most rudimentary level. "If we can understand what is going on at the nanoscale, we can better design materials with improved properties," underlines Holmestad. The ideal device for studying materials at the atomic scale is the transmission electron microscope.

TEM allows researchers to elucidate a material's crystal structure, view how its atoms are organised, find the composition, examine irregularities and even measure the bonds between individual atoms. Combined with the use of cutting-edge quantitative analysis techniques and advanced computational tools, Holmestad's transmission electron microscopes have been able to decipher the crystal structures of most precipitates in aluminium alloys. Without this technique, it would be impossible to see the link between the nanoscale physics and the resulting real-world functions of the aluminium alloys under investigation.

3D VIEWING

Another methodology that has proven pivotal



Holmestad's team works within the red box, which includes precipitates, clustering and phase transformations.

INTELLIGENCE

FUNDAMENTAL INVESTIGATIONS OF PRECIPITATION IN THE SOLID STATE WITH FOCUS ON AL-BASED ALLOYS

OBJECTIVE

Using transmission electron diffraction and microscopy to study the microstructure of materials, in particular precipitates in age-hardenable aluminum alloys, and the relationship with macroscopic properties.

KEY COLLABORATORS

Professor Knut Marthinsen; **Dr Sigurd Wenner**; **Dr Flemming Ehlers**, Norwegian University of Science and Technology (NTNU)

Dr Calin Marioara; **Dr Sigmund Andersen**, SINTEF Materials and Chemistry

Dr Trond Furu; **Dr Jostein Røyset**; **Dr Oddvin Reiso**; **Dr Takeshi Saito**, Norsk Hydro, Norway

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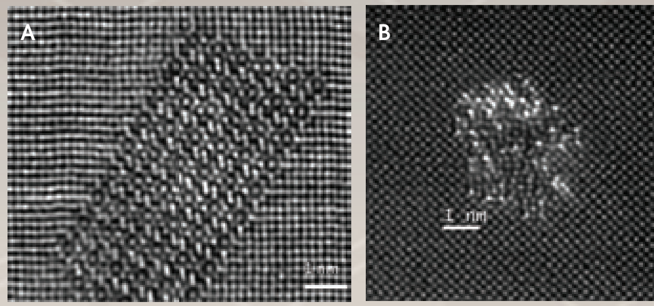
RANDI HOLMESTAD has been Professor in the Department of Physics at NTNU, Norway, for 15 years. After receiving a Master's degree in Technical Physics at NTNU in 1991, she became a DrIng in Physics in 1994. Over the years, she has published 150 papers and taken part in several projects, including at Arizona State University and the University of Illinois. An advisor to SINTEF Materials and Chemistry, her research on precipitation in aluminium alloys using electron microscopy includes a number of projects in collaboration with SINTEF and Norsk Hydro, Norway.



A. Ordered precipitate without Cu. **B.** Disordered precipitate with Cu. In both images, the aluminium host lattice can be seen around the precipitate. HAADF-STEM images taken by Sigurd Wenner, Takeshi Sato and Calin Marioara.

for precise microstructure determination is APT. APT is particularly helpful in the characterisation of small clusters and precipitates in metallic alloys as it uses an atom probe which aids atom real-space mapping in 3D with near-atomic resolution. The Norwegian research group has been working with Rouen University in France on APT studies. APT has found particular use in the early stages of precipitation where TEM cannot be used. APT and TEM are complementary – APT can be used to study composition and TEM can add information about atomic column positions and crystal symmetries. "It's actually really fun that, by studying almost single atoms, we can help the Norwegian aluminium industry in their production of tonnes of aluminium!"

Simultaneous to experiments, the researchers have also been conducting DFT simulations. "With DFT we can check, on the atomic scale, the energy needed to form different phases and tell which one is more likely to form," explains Holmestad. "Also, simulations of TEM image intensities and quantitative comparisons between experimental and simulated images have been very useful in our studies."

WHEEL OF FORTUNE

This combination of techniques is already yielding results. A recent example concerns the common Al-Mg-Si alloy with a small amount of Cu. Normally, its strength disappears when left above 200 °C for some tens of hours. The reason for this is that the precipitates transform to other phases which are not hardening. "In some applications – for example, steering columns in cars – it is of pivotal importance to maintain strength at these (relatively high) temperatures," Holmestad adds.

Crucially, the NTNU-SINTEF-Norsk Hydro team has a patent for a specific alloy composition with a higher Mg:Si ratio and the addition of a very small amount of other elements, such as germanium and silver, which maintains strength at higher temperatures.

ALUMINIUM TO ORDER

More generally, Holmestad and colleagues have concluded that strength is very dependent on the type and size of precipitates, and that these can be manipulated by varying the two primary alloy parameters: composition and heat treatment. Consequently, the research group aims to be able to create 'alloys by design' via in-depth knowledge garnered through their fundamental physics investigations.

To obtain specific properties, industry has traditionally employed trial and error, but the increased understanding Holmestad's team provides should make this a thing of the past: "Given that we understand the physics responsible for nucleation and precipitation, and all the processes down to the atomic scale, we can provide industry with input so that desired properties can be achieved without too much testing, maybe also optimised in better ways to obtain new improved alloys designed for a certain use".

RECYCLE AND UPGRADE

Holmestad and her group also wish to utilise their combination of techniques to improve the overall properties of recycled aluminium. 75 per cent of all aluminium alloys ever produced are still in use. By recycling, just 5 per cent of the energy needed to produce alloys from scratch is consumed. These savings understandably make recycling alloys a priority for aluminium companies and their customers. However, while recycling is efficient with regards to resources and obviously beneficial for the environment, impurities and trace elements are costly to eradicate and can affect the microstructure of the final material.

"TEM can help us to study the microstructure and find out how extra impurities from recycling will affect the alloy," explains Holmestad. "If we understand this, we can make 'alloys by design'." Therefore, through nanoscale understanding, the group aims to not only deal with the extra elements in recycled aluminium so that negative properties do not surface, but actually utilise them to obtain improved properties.

This and other projects currently being pursued by the Norwegian group – including various studies in international collaboration with several research teams in Japan – will ensure research on alloys and customising them continues to progress. And by expanding knowledge of precipitates and how their potential can be optimised for specific purposes, Holmestad's objective of 'alloys by design' may be reached sooner than most would expect.