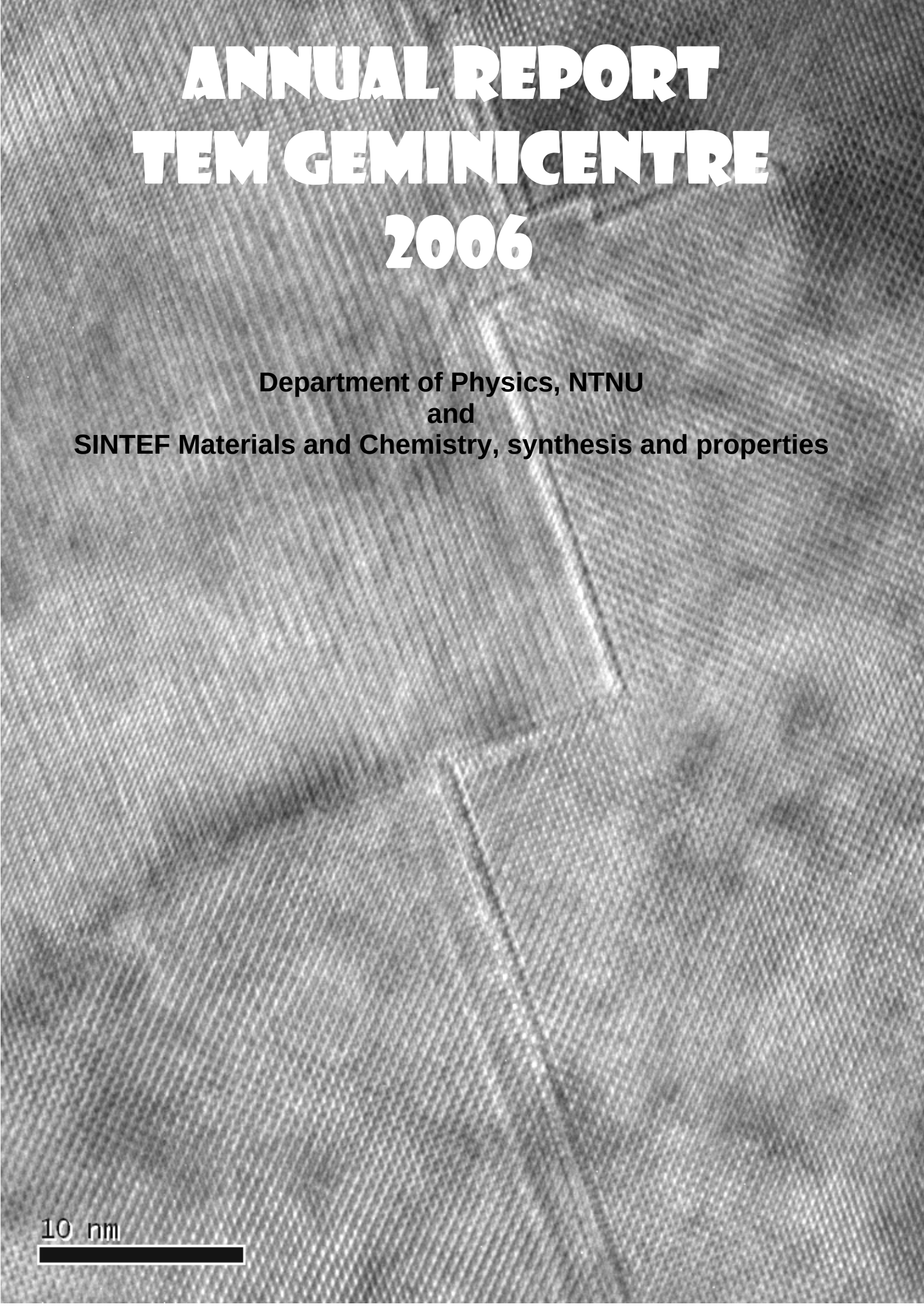
ANNUAL REPORT TEM GEMINICENTRE 2006

Department of Physics, NTNU
and
SINTEF Materials and Chemistry, synthesis and properties



Personnel, working in the group during 2006

Randi Holmestad (professor NTNU)
Bård Tøtdal (Associate professor NTNU)
John Walmsley (SINTEF Scientist, Professor II NTNU)
Sigmund J. Andersen (SINTEF scientist)
Calin Marioara (SINTEF scientist)
Børge Forbord (SINTEF scientist)
Bjørn Steinar Tanem (SINTEF scientist)
Heimir Magnusson (Engineer NTNU)
Ton van Helvoort (Postdoc NTNU, SINTEF scientist)
Rene Vissers (postdoc NTNU)
Per Erik Vullum (Postdoc NTNU)
Anders Frøseth (NTNU scientist, part time)
Wakshum Mekonnen Tucho (PhD student NTNU)
Heidi Nordmark(PhD student NTNU)
Håkon Stokka Hasting (PhD student NTNU)
Roya Dehghan (PhD student NTNU)
Ragnhild Sæterli (Master student, PhD student, NTNU)
Bjørn Gunnar Soleim (scientific assistant NTNU, part time)
Asgeir Birkeland (Master student NTNU)
Åsmund Monsen (Master student, summer job NTNU)
Elin Stubhaug (Master student NTNU)
Inger Marie Skoe (Master student NTNU)
Maulid Mohamed Kivambe (Master student NTNU)
Espen Eberg (Master student, IET, NTNU)
Suresh Prasad Gupta (Master student NTNU)
Kim Vidar Bakken (Master student NTNU)
Liviu Holt (project student NTNU)
Malin Torsæter (project student NTNU)
Åsmund Almli (project student NTNU)
Anton Nordstrøm (project student NTNU)

John, Randi, Heimir and Ton on the 2010F

The front page is a HREM pattern of $SrRuO_3$, taken by A.T.J. van Helvoort

TEM Geminicentre

The Geminicentre in TEM was created April, 19. 2006. An article from SINTEFs web page is shown under. An official opening of the Geminicentre will take place in 2007.

NTNU og SINTEF satser sammen på avansert mikroskopi

SINTEF og NTNU formaliserer sitt samarbeid innenfor transmisjonselektronmikroskopi. Fagfeltet er et viktig hjelpemiddel innen nanoteknologi og ved utvikling av nye materialer.

Konsernsjef Unni Steinsmo i SINTEF gratulerer NTNU-professor Randi Holmestad og SINTEF's John Walmsley med etableringen av det nye Gemini-senteret. Foto: Geir Otto Johansen.

De to institusjonene har gått sammen om å opprette et såkalt Gemini-senter innen transmisjonselektronmikroskopi (TEM) – det 17. i rekken av slike sentre.
I et Gemini-senter har de samarbeidende fagmiljøene felles strategiprosesser. Partene koordinerer også arbeidet med søknader om større forskningsprosjekter og -programmer. Sentrene arbeider i tråd med visjonen: "Være internasjonalt fremragende sammen".
I det nyetablerte senteret inngår NTNUs Institutt for fysikk og avdelingen Syntese og egenskaper ved SINTEF Materialer og kjemi.

Milliontedels millimeter

Det nye senteret disponerer tre TEM – avanserte "forstørrelsesglass" som bruker elektronstråler til å "se" inn i materialers indre og deres overflater.
Flaggskipet er et feltemisjons TEM, et instrument som har en rekke analysemetoder med nanometers oppløsning (1 nanometer = 1 milliontedels millimeter). I tillegg kan det "se" inn i materialene på atomnivå.
TEM brukes til å bestemme sammenhengen mellom materialers struktur og deres egenskaper, og er et viktig hjelpemiddel ved utvikling av nye materialer. Et klassisk eksempel: TEM gjør det mulig å se utfellinger i aluminium. Bildene viser hvordan atomer fra de tilførte fremmedmetallene i aluminiumlegeringer klumper seg sammen i bittesmå "nåler". "Nålene" blir en slags armering i materialet, og mikroskopbildene bidrar med kunnskap som gjør det mulig å skreddersy denne armeringen.

Mange bruksområder

NTNU og SINTEF bruker også TEM til å studere aluminiumoverflater, nyutviklede membraner, materialer for hydrogenlagring, plast- og plastkompositter, biomaterialer, tynnfilm for elektronikkanvendelser, nanopartikler og solcellematerialer pluss keramiske materialer for bruk i brenselceller.

Dette mikroskopet, et feltemisjons TEM, som her bemannes av NTNUs Randi Holmestad og SINTEFs Bjørn Steinar Tanem, er det nye Gemini-senterets flaggskip. Foto: Svein Tønseth

Øker slagkraften

TEM-miljøet i Trondheim har en stor kontaktflate mot andre fagmiljøer nasjonalt – både innenfor NTNU/SINTEF-systemet, mot industri og instituttsektoren. Miljøet har også utstrakt internasjonalt samarbeid.

– Senteretableringen gir en verdifull styrking av det eksisterende samarbeidet mellom NTNU og SINTEF på dette fagfeltet, sier fysikkprofessor Randi Holmestad ved NTNU.

– Slagkraften i et forskningsmiljø står og faller med antallet hoder. Ved NTNU utdanner vi forskere som forsvinner etter to til tre år med doktorgrads- eller post.doc-studier. Samarbeidet med SINTEFs forskere sikrer kontinuitet i forskningen, sier Holmestad.

Forsker Bjørn Steinar Tanem ved SINTEF Materialer og kjemi påpeker at senteretableringen er viktig for hjemhenting av kunnskap til Norge:

– Når SINTEF og NTNU posisjonerer og profilerer seg sammen på denne måten, blir det lettere å få gjennomslag når vi søker om deltakelse i store internasjonale prosjekter, sier Tanem.

Av Svein Tønseth

Scientific highlights

2006 has again been a very productive year for the TEM Geminicentre, as shown by the publication list below. We have built up competence and skills in several new techniques and materials. The TEM Geminicenter has many collaborators at several departments at NTNU, within SINTEF and internationally, working on a variety of materials. Here follow a few examples.

Ferroelastic nanotwins

Oxide perovskite materials with properties tailored by chemical substitution have been extensively studied due to novel electrical and magnetic properties. Ferroelastic properties may also be tailored by chemical substitution, and here we report the formation of two different types of ferroelastic domains in substituted $LaCoO_3$-based materials. In addition to deformation twinning resulting in domains with width ~20-200 nm, a novel type of nano-twinned ferroelastic domains becomes more abundant at high substitution levels. These domains have a one-dimensional superstructure corresponding to doubling or tripling of the pseudo cubic unit cell due to twinning at the atomic scale. Thus twinning occurs on two different length scales leading to two different domain types. Both types show ferroelastic properties, but they have different domain reorientation characteristics. The coexistence of deformation twinning and nano-twinned domains represent new and advanced mechanisms for accommodation of epitaxial film-substrate mismatch in materials such as magnetoresistant $La_{1-x}Sr_xMnO_3$ and multiferroic $BiFeO_3$.

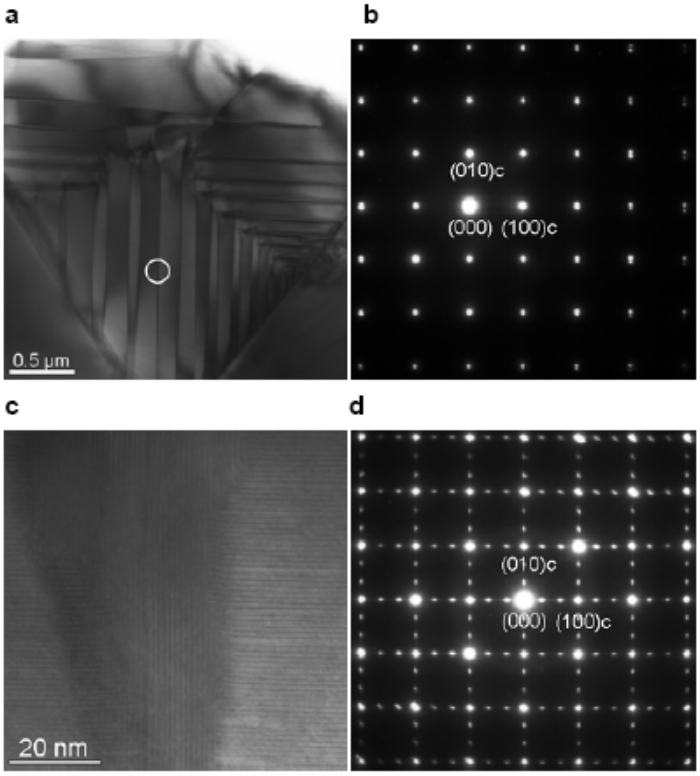


Figure 1. Two types of domains in $LaCoO_3$ observed with TEM. a) (100) deformation twins in two perpendicular pseudo cubic [100]-directions in $LaCoO_3$. b) Electron diffraction from the circular marked area in a) and viewed down along the hexagonal $[4\bar{2}1]$ direction, corresponding to the pseudo cubic [001] direction. c) Two nano-twinned domains in $LaCoO_3$ showing a superstructure with a periodicity of $3a_c$. d) Electron diffraction from an area with several nano-twinned domains, confirming the tripling of the pseudo cubic unit cell along the [100] and [010] directions.

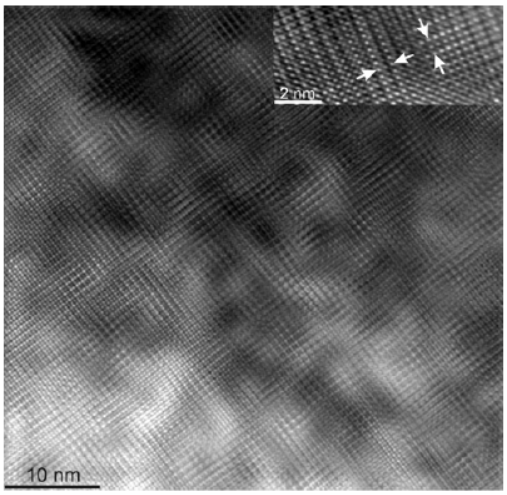
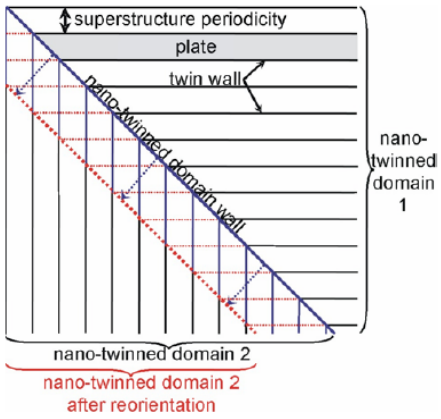


Figure 2. Nano-twinned domains in $La_{0.5}Sr_{0.5}Fe_{0.5}Co_{0.5}O_{2.905}$. The inset shows a magnified area consisting of two nano-twinned domains. The arrows point out the doubling of the pseudo cubic lattice parameter.



Scheme 1. Illustration of two nano-twinned domains separated by a domain wall. Each domain consists of 2-3 cubic unit cell thick plates separated by twin walls. During compression, the nano-twinned domain wall will propagate in order to increase the size of the domain with the most favourable orientation with respect to the mechanical field. The thickness of the plates remains constant while the domain size changes. The volume reoriented due to the movement of the nano-twinned domain wall results in reorientation of the ordering in volume marked by the blue (initial) and red (final) position of the nano-twinned domain wall.

From
Vullum PE, Holmestad R, Lein HL, Mastin J, Einarsrud MA and Grande T, *Ferroelastic nano-twinned domains in $LaCoO_3$-based perovskites*, Submitted 2006.

Cu-containing precipitates in Al alloys

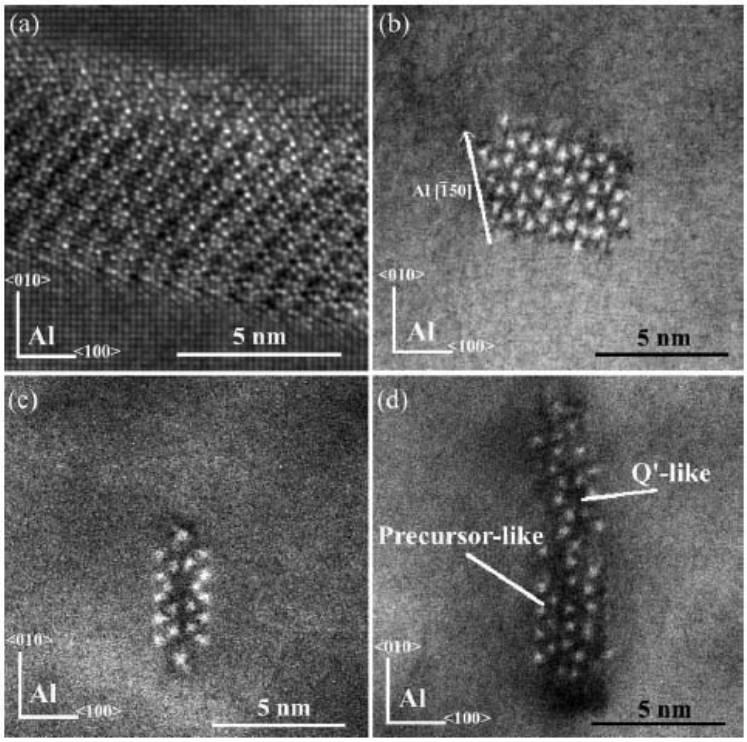


Figure 3. Precipitates found in matrix in the over-aged condition: (a) HREM image of Q′ phase; (b) Z-contrast image of Q′ phase; (c) Z-contrast of precursor-phase aligning in Al ⟨100⟩; (d) Z-contrast of mixed phase with both precursor-like and Q′ phase-like elements.

High-resolution annular dark field imaging has been used to study small precipitates in the Al matrix of 6XXX-series aluminium alloys. Cu-containing columns in precipitates were imaged by atomic number contrast along [100] matrix direction. At an under-aged condition, large, lath-shaped particles of the Cu-containing Q'-phase were observed at grain boundaries. At over-aged

conditions, Q' was the main matrix phase. In both cases the laths showed an Al {150} habit plane. A precursor phase was found, with different arrangement of Cu-rich columns from that of the Q'-phase and sharing Al {100} habit planes. Precipitates containing elements of both the Q'-phase and the precursor were found to be common. Annular dark field imaging can differentiate directly between phases. The method complements and aids interpretation of conventional high-resolution TEM images and nano beam diffraction observations. The results obtained contribute to an improved understanding of the crystallography of the precursor and details of the structural transformation taking place during the precipitation process.

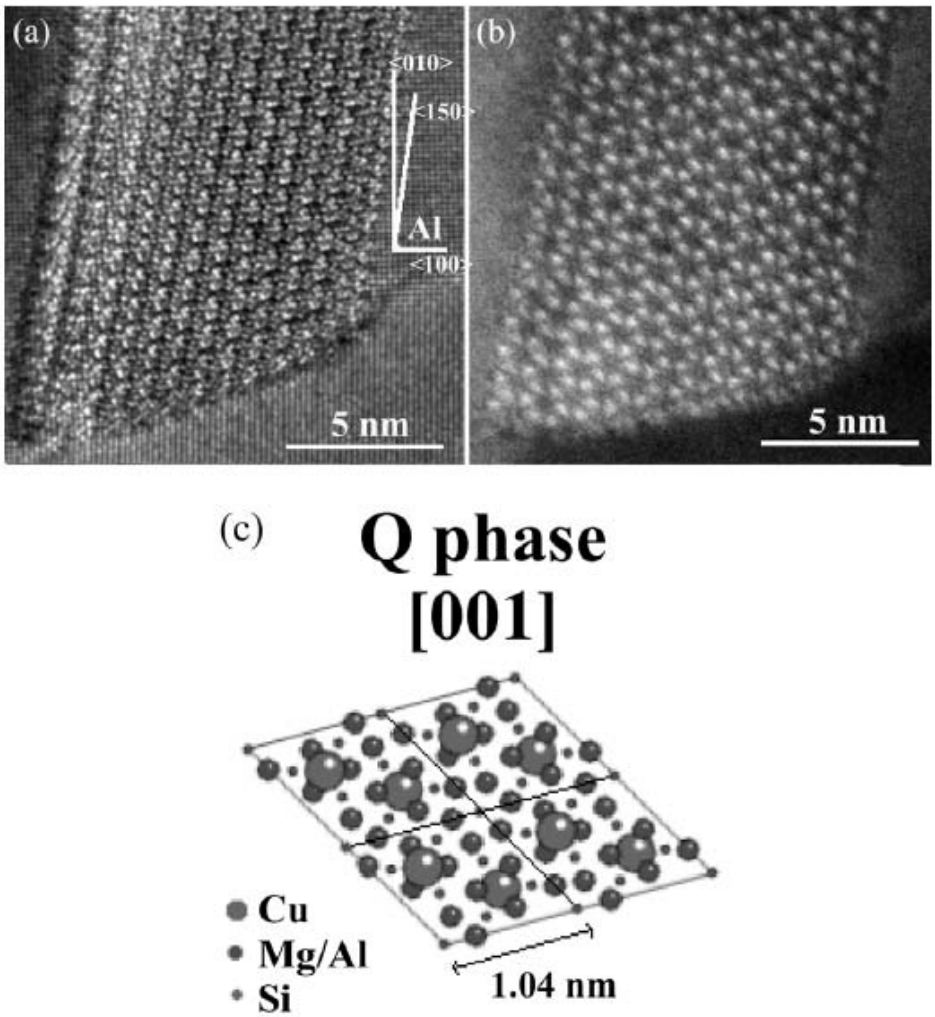


Figure 2. Cross-section of lath-shaped Q′-phase at grain boundary directed along an Al⟨100⟩||Q′[001] direction. The Q′-phase has nucleated on grain boundary in the under aged condition: (a) HREM; (b) ADF STEM image showing Cu-containing atomic columns as bright spots; (c) Q-phase model in [001]-orientation (2 × 2 unit cells), as given by Arnberg and Aurivillus [8].

From:
Hasting HS, Walmsley JC, van Helvoort ATJ, Marioara, CD, Andersen S, Holmestad R. *Z-contrast imaging of the arrangement of Cu-precipitates in 6xxx-series aluminium alloys,* Philosophical Magazine Letters, 86, 589-597, 2006

Lead segregation in Al surfaces

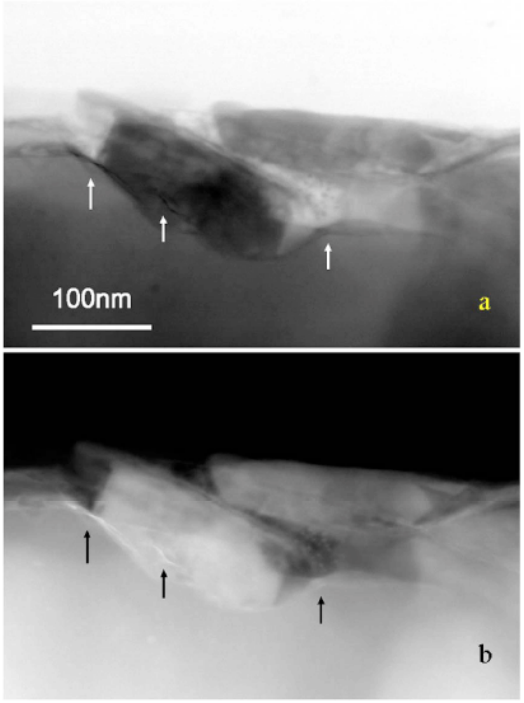


Figure 5. STEMs of the thermal oxide surface in cross section orientation. (a) Bright field image and (b) dark field image. Close examination suggests the presence of a thin film of high-density material that gives dark contrast in (a) and bright contrast in (b).

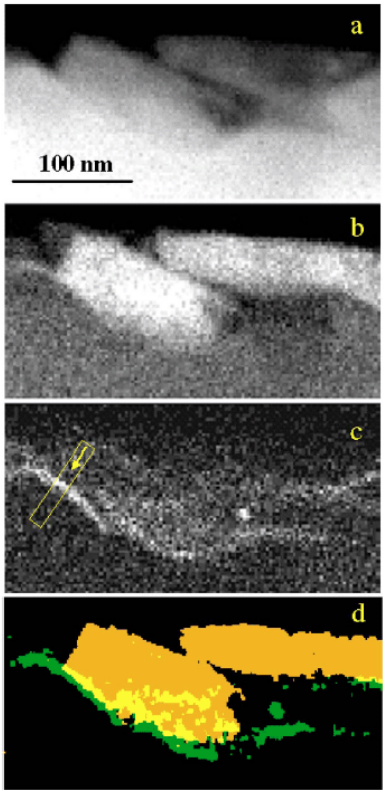


Figure 6. (Color online) EDS maps from the same area as Fig. 5. (a) Aluminum, (b) oxygen, (c) Pb, and (d) a combination of the three elements.

The segregation of Pb on model binary AlPb alloys, containing 20 and 50 ppm Pb, as a result of heat treatment in air at 600°C and its influence on electrochemical properties have been studied. Enrichment of metallic Pb, concentrated towards the oxide side of the oxide-metal interface, was confirmed by X-ray photoelectron spectroscopy. Transmission electron microscopy revealed a nearly continuous nanometer-scale Pb film at the oxide-metal interface. Significant anodic activation of the AlPb alloy surface in relation to pure Al in chloride media is attributed to the Pb film destabilizing the thermal oxide. The degree of activation is limited by the surface coverage of the film, and discrete Pb particles in the oxide do not contribute to the activation. After initiation at certain grain boundaries and discrete sites on grain bodies, corrosion in the active state spread nearly two dimensionally as the Pb film on the corroded sites was destroyed as a result of corrosion, and corroded sites repassivated. The formation of the γ-Al_2O_3 thermal oxide during heat treatment is thus crucial in the formation and existence of the Pb film wetting the metal surface.

From:
Walmsley JC, Sævik Ø, Graver B, Mathiesen RH, Yu Y and Nisancioglu K *Nature of Segregated Lead on Electrochemically Active AlPb Model Alloy*, Journal of the Electrochemical Society, 154 (1) C28-C35, 2007.

Ti enhanced reversible hydrogen storage

A reliable method for reversible hydrogen storage is the key requirement for large scale application of proton exchange membrane fuel cells in cars. Among the various approaches for hydrogen storage, complex hydrides have shown to be the most promising group of materials. Until recently, complex hydrides were not considered as potential materials for reversible hydrogen storage due to the high stability of the materials at operating conditions for fuel cells. This picture was altered when it was discovered that adding transition metals, such as Ti, to sodium alanate ($NaAlH_4$) enabled reversible hydrogen absorption at moderate temperatures. In addition, the kinetics of hydrogen reabsorption were increased by several orders of magnitude. Since the discovery of adding Ti as a catalyst to $NaAlH_4$, numerous research groups have tried to optimize the parameters involved in reversible hydrogen storage with respect to kinetics and storage capacity. However, further optimization depends on a thorough understanding of the fundamental question: What are the state, location and role of the Ti catalyst during reversible hydrogen storage in $NaAlH_4$.

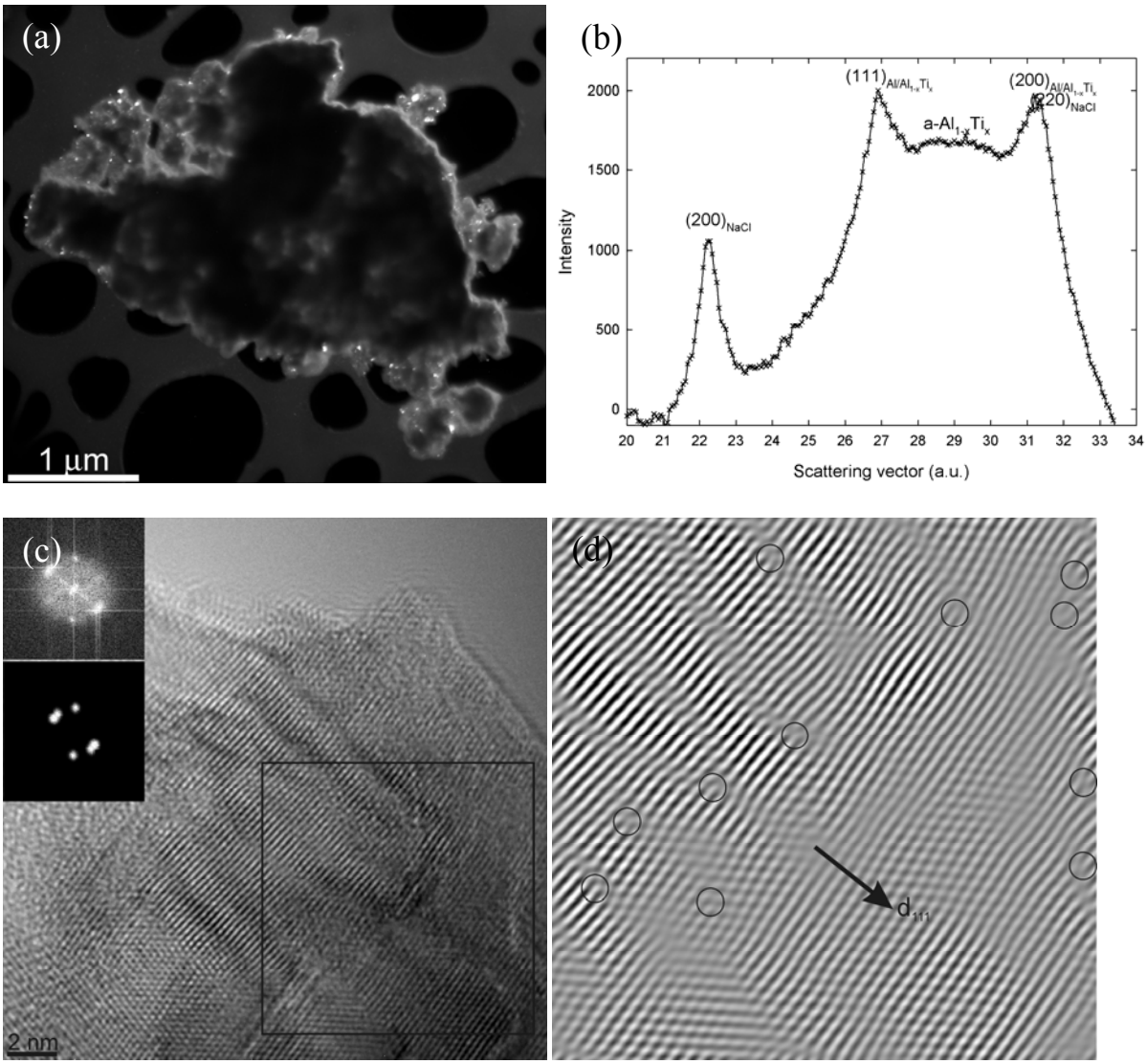


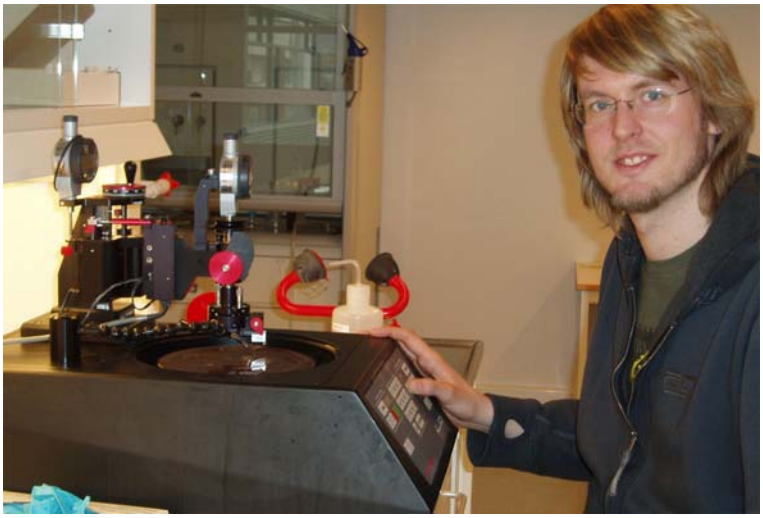
Figure 1. (a) Dark field image showing the dispersion of small $Al_{1-x}Ti_x$ crystallites on the surface of the alanate grains. (b) Integrated electron diffraction pattern which shows the $Al_{1-x}Ti_x$ primary amorphous halo between the crystalline Al and $Al_{1-x}Ti_x$ (111) and (200) diffraction lines. (c) $Al_{1-x}Ti_x$ particle embedded in the $NaAlH_4$ surface. (d) The filtered inverse Fourier transform image of the framed area in (c) demonstrates the high density of (111) edge dislocations.

A combination of transmission electron microscopy and synchrotron X-ray diffraction has been used to prove that the Ti catalyst is located on the surface of the alanate grains. Further, it has been shown that the Ti catalyst exists as nanoscopic surface particles in a combination of an amorphous and a crystalline $Al_{1-x}Ti_x$ state. The small $Al_{1-x}Ti_x$ crystallites are heavily defected with numerous dislocations. The very well dispersed and densely dislocated $Al_{1-x}Ti_x$ surface particles are believed to be the main reason why Ti has shown superior catalytic properties compared to other transission metals.

From:
Vullum PE, Pitt MP, Walmsley JC, Hauback B, Holmestad R. *Transmission electron microscopy characterization of $NaAlH_4$,* to be submitted.

Instruments

The group has 3 TEMs -a Jeol 2010F (2003), a Philips CM30 (1990) and a Philips EM400T. The 2010F offers many techniques, and during the last year we have paid particular attention to applying developments of the STEM (scanning transmission electron microscopy) technique, EELS (electron energy loss spectroscopy) and EFTEM (energy filtered transmission electron microscopy) and to make their applications user friendly. We have invested in a gas transfer reaction holder for catalysis and hydrogen storage material work, as well as a tomography holder for 3 dimensional imaging. The group has continued to make progress with new applications of sample preparation techniques, such as tripod polishing and ultramicrotomy.



Espen has used many hours doing tripod polishing

Miscellaneous

The group educated 1 PhD and 5 Master candidates in 2006. Several members of the group participated in international conferences and meetings. A PhD course in Electron Microscopy and diffraction was given for 6 PhD students. The group has had lunch meetings every second Friday, and have given many guided tours for high school students.

Ragnhild giving a demo of the TEM for high school students

The TEM group has a bright future. We will get a new permanent position at NTNU in 2007. We have 4 diploma students in the first semester of 2007. Several new projects and activities starting in 2007 have been secured. These include two large projects on Aluminium; a large industry initiated project (BIP) chaired by Hydro Aluminium on nucleation control, and a more fundamental (FRINAT) project on precipitation. These will involve several new PhD students, Postdocs and several man-years of SINTEF activity over the next 5 years. Two large and highly successful Norlight projects, Light Metal Surface Science and Heat Treatment Fundamentals finished during 2006. Both of these have included a great deal of Geminicentre activity which has involved significant interaction with other NTNU and SINTEF groups and Norwegian Industry which will continue into the future.

External projects in 2006

Larger projects are listed by funding type, name, source of funding, duration, collaborators and funding level for 2006. Many of these projects involve both NTNU and SINTEF activity.

SUP: Micro- and nanostructural based materials developments (NFR; 2000-2007) Dept. of Materials Technology and Engineering, ~ 1000 KNOK

KMB: Heat treatment fundamentals (NFR, Hydro, Elkem; 2000-2006), Dept. of Materials Technology and Engineering, ~ 4400 KNOK

KMB: Light metal surface Science (NFR, div. industry; 2000-2006) Dept of Materials Technology and Engineering, ~ 100 KNOK

KMB: Advanced electron microscopy of catalysts (NFR, Statoil, 2006-2010) Dept. of Chemical Engineering, ~ 800 KNOK

NANOMAT: Membranes for hydrogen separation (NFR, 2003-2007) SINTEF Oslo, Dept. of Chemical Engineering, ~ 650 KNOK

FRINAT: Studies of the electronic structure at the nanoscale (NFR, 2006-2009), University of Oslo, ~ 650 KNOK

NANOMAT: Low dimensional electroceramics (NFR, 2003-2007), Dept. of Telecom. and electronics, ~ 70 KNOK

BIA: Kimdanningskontroll for optimaliserte egenskaper (NFR, Hydro, SteerTec, 2006-2011) ~500 KNOK

Numerous smaller projects, both academic and industrial have been carried out in addition.

Publications Geminicentre TEM, 2006

19 journal papers

Andresen M, Johansson LS, **Tanem BS**, Stenius P, *Properties and characterization of hydrophobized microfibrillated cellulose,* Cellulose 13, 665-677, 2006.

Arstad B, Venvik, H, Klette H, **Walmsley JC**, **Tucho WM**, **Holmestad R**, Holmen A and Bredesen R *Studies of self-supported 1.6 µm Pd/23 wt.% Ag membranes during and after hydrogen production in a catalytic membrane reactor*, Catalysis today, 118, 63-72, 2006.

Forbord B, Auran L, Lefebvre W, Hallem H, Marthinsen K, *Rapid precipitation of dispersoids during extrusion of an Al-0.91 wt.% Mn-0.13 wt.% Zr-0.17 wt.% Sc-alloy* Materials Science and Engineering A, 424, 174-180, 2006.

Graver B, **van Helvoort A**, **Walmsley JC**, Nisancioglu K *Surface segregation of indium by heat treatment of aluminium*, Materials Science Forum 519-521, 673-678, 2006.

Hallem H, Rittel WJ, **Forbord B**, Marthinsen K *Recrystallisation resistance of extruded and cold rolled aluminium alloys with additions of Hf, Sc and Zr*, Materials Science Forum 519-521, 525-530, 2006.

Hallem H, Lefebvre W, **Forbord B**, Danoix F, Marthinsen K, *The formation of Al-3(ScxZryHf1-x-y)-dispersoids in aluminium alloys,* Materials Science and Engineering A 421, 154-160, 2006.

Hasting HS, Walmsley JC, Marioara CD, van Helvoort ATJ, Holmestad R, Danoix F, Lefebvre W, *Characterisation of early precipitation stages in 6xxx series aluminium alloys,* Journal of Physics, Conference Series, 26, 99-102, 2006.

Hasting HS, Walmsley JC, van Helvoort ATJ, Marioara, CD, Andersen S, Holmestad R. *Z-contrast imaging of the arrangement of Cu-precipitates in 6xxx-series aluminium alloys,* Philosophical Magazine Letters, 86, 589-597, 2006.

van Helvoort, ATJ, Tanem, BS, Holmestad, R, *Annular bright and dark field imaging of soft materials.* Journal of Physics, Conference Series, 26, 42-45, 2006.

Knowles KM and **van Helvoort ATJ**, *Anodic bonding*, International Materials Review, 51, 273-311, 2006.

Larsen MH, **Walmsley JC**, Lunder O, Nisancioglu K *Significance of low copper content on grain boundary nanostructure and intergranular corrosion of AlMgSi(Cu) model alloys* Materials Science Forum, 519-521, 667-671, 2006.

Lein HL, Andersen OS, **Vullum PE**, Lara-Curzio E, **Holmestad R**, Einarsrud MA, Grande T *Mechanical properties of mixed conducting La0.5Sr0.5Fe1-xCoxO3-delta (0 <= x <= 1) materials***,** Journal of solid state Electrochemistry 10, 635-642, 2006.

Marioara CD, **Nordmark H**, **Andersen SJ** and **Holmestad R**, *Post-beta" phases and their influence on microstructure and hardness in 6xxx Al-Mg-Si alloys*, Journal of Materials Science 41, 471-478, 2006.

Nordmark H, Ulyashin A, **Walmsley JC, Tøtdal B** and **Holmestad R,** *Evolution of hydrogen induced defects during annealing of plasma treated Czochralski silicon,*Nuclear Instruments and Methods in Physics Research B 253, 176–181, 2006.

Nes E, Holmedal B, **Forbord B**, *The effect of boundary structure on the mechanical properties of aluminium alloys,* Materials Science Forum 519-521, 63-70, 2006.

Sandberg N and **Holmestad R**, *First principles calculation of impurity diffusion barriers in Al* Physical Review B, 73, Art. No. 014108, 2006.

Svenningsen G, Larsen MH, **Walmsley JC**, Nordlien JH and Nisancioglu K, *Effect of artificial aging on intergranular corrosion of extruded AlMgSi alloy with small Cu content*, Corrosion Science 48, 1528-1543, 2006.

Tanem, BS, Kvien, I, **van Helvoort, ATJ,** Oksman, K, *Morphology of Cellulose and its Nanocomposites*, Cellulose Nanocomposites; Processing, Characterization and Properties. Washington: American Chemical Society, 48-62, ISBN 0-8412-3980-0, 2006.

Vullum PE, Mastin, J, Wright J, Einarsrud MA**, Holmestad R** Grande T, *In-situ synchrotron X-ray diffraction of ferroelastic $La_{0.8}Ca_{0.2}CoO_3$ ceramics during uniaxial compression*, Acta Materialia 54, 2615-2624, 2006.

12 accepted journal papers

Andersen SJ, Marioara CD, Vissers R, Frøseth AG, Zandbergen HW, *The structural relation between precipitates in Al-Mg-Si alloys, the Al-matrix and diamond silicon, with emphasis on the trigonal phase U1-$MgAl_2Si_2$*, Accepted, Mat. Sci. Eng .A.

Chytil S, Glomm WR, Kvande I, Zhao T, **Walmsley JC** and E. Blekken E, Platinum incorporation into the SBA-15 mesostructure via deposition-precipitation method: Pt nanoparticle size estimation and catalytic testing, Accepted, Topics in Catalysis.

Hasting HS, Lefebvre W, **Marioara CD**, **Walmsley JC**, **Andersen SJ**, **Holmestad R** and Danoix F *Comparative study of β''in 6xxx Al-alloy with Atom probe and HRTEM,* Accepted, Surface and Interface Analysis.

Huber F, Yu Z, **Walmsley JC**, Venvik H, Holmen A, *Nanocrystalline Cu-Ce-Zr mixed oxide catalysts for clean fuel applications: Carbon nanofibers as dispersant for the mixed oxide particles*, Accepted, Applied Catalysis B: Environmental.

Iojoiu EE, Miachon S, Landrivon E, **Walmsley JC**, Raeder H, Dalmon JA, Wet air oxidation in catalytic membrane reactor: model and industrial wastewaters in single tubes and multichannel contactors, Accepted, Applied Catalysis B: Environmental.

Mottern ML, Tyholt F, Ulyashin A, **van Helvoort ATJ**, Verwey H and Bredesen R, *Textured ITO thin films by chemical solution deposition and rapid thermal processing*, Accepted, Thin Solid films.

Ochoa-Fernández E, Lacalle-Vilà C, Christensen KO, **Walmsley JC**, Rønning M, Holmen A and Chen D, *Ni Catalysts for Sorption Enhanced Steam Methane Reforming*, Accepted, Topics in Catalysis.

Vissers R, van Huis MA, Jansen J, Zandbergen HW, **Marioara CD, Andersen SJ,** *The Crystal Structure of theβ' Phase in Al-Mg-Si Alloys* Accepted, .Acta Mater.

Vullum PE, **van Helvoort ATJ**, Mastin J, Andersen ØE, Einarsrud MA, Grande T, **Holmestad R**, *Grain boundary analysis and secondary phases in LaCoO3-based perovskites*, Accepted, Journal of Materials Science

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