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Danish Chinese Center for Nanometals - Annual report 2011

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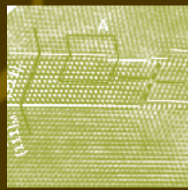

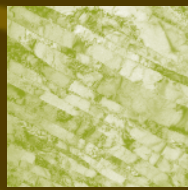

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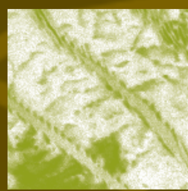
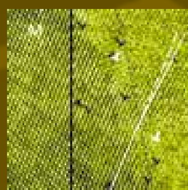
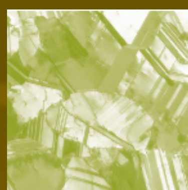




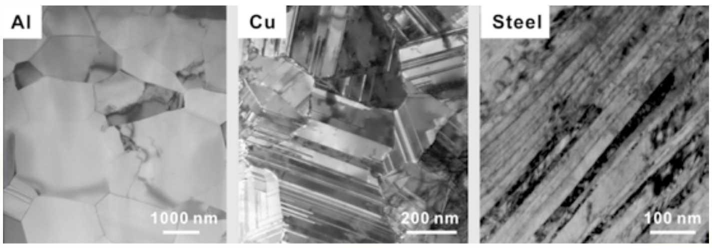


Danish – Chinese Center for Nanometals

Nanometals

The Center investigates metals, including alloys, with internal length scales ranging from a few nanometers to a few micrometers. These are termed nanometals, and have new and interesting properties, such as exceptional mechanical strength, which are related to their internal structure. The scientific focus is to understand and control the mechanisms and parameters determining the mechanical and physical properties of such nanometals as well as their thermal stability.

The internal structure of nanometals is highly diverse, consisting of different types of internal interfaces, spatially arranged in different morphologies, as exemplified by equiaxed grains in aluminum, lamellar nanotwins within equiaxed sub-micrometer grains in copper, and lamellar boundaries between two phases in steel. The morphology as well as the chemical composition of nanometals can be manipulated and are key features to be optimized for future applications of nanometals.



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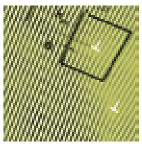
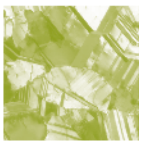
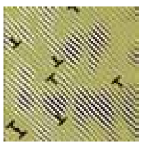


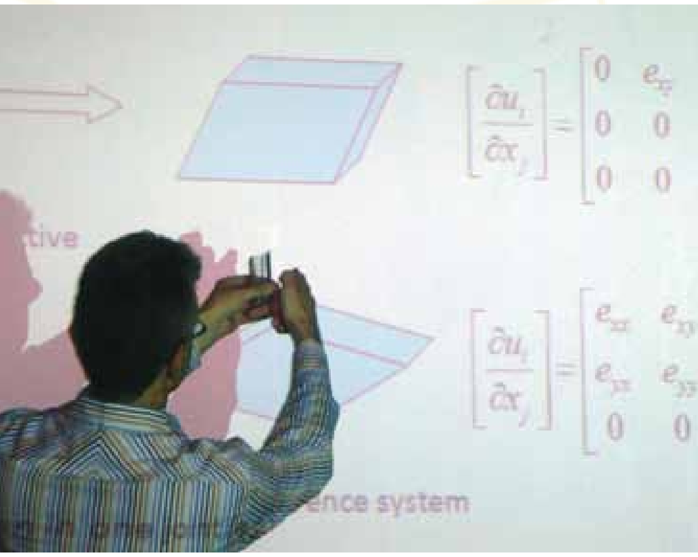




Danish – Chinese Center for Nanometals

Introduction

Our Danish-Chinese Center for Nanometals is now 2 ½ years old, and we are happy to report that the original vision of both synergy and complementarity in a joint Danish-Chinese research collaboration has been successful. Also the personal connections between Danish and Chinese scientists and students have been strengthened significantly, and in many ways we feel like one big Danish-Chinese scientific family.



The scientific highlights of 2011 are many. We have therefore changed the format of this report to a certain degree compared to previous years to make room for eight highlights. These encompass the key scientific themes of our center and deal with:

(i) advanced characterization of mechanical properties, including quantification of local effects and nano-mechanical testing in a transmission electron microscope;

(ii) development and tailoring of nanostructures to optimize mechanical properties and thermal stability. A new mechanism has been discovered based on triple junction motion, which can account for the hitherto unexplained observation by many scientists of annihilation of deformation induced boundaries;

(iii) effects of solutes and precipitates on the mechanical properties and thermal behavior of metals and alloys which take advantage of knowledge obtained for model materials to analyze phenomena in industrial alloys.

Another highlight of 2011 was the Summer School held in Beidaihe, China, during the week August 14-18th. The theme of the Summer School was Nanostructured Metals for Energy, and was organized with 3 days of lectures by leading international scientists outside the Center and 2 days of presentations by scientists and students from the Center on ongoing Center projects. In addition two poster sessions were held, with a total of 36 posters covering all aspects of the work within the center. The discussions at the poster sessions were so vivid that even the long period of 4 hours set aside for the poster sessions was too short so that the discussions continued during many of the following breaks. Altogether the Summer School contributed to strengthening the scientific and personal contacts between the Center participants both juniors and seniors.

At the Summer School all the senior partners had a fruitful meeting discussing the future plans for a possible extension of our Center for another 3 years from September 2012. The agreed new plan builds on the successes of the present first 3 years, with an increased focus on graded structures, in particular structures with hard wear resistant surfaces, and on effects of solutes and precipitates on mechanical properties and thermal stability of nanometals. Both these themes are of upmost importance for practical applications, and a key aim of the extended Center period is to use our basic understanding of nanometals to promote the industrial use of this class of very powerful metals. As an example hard and wear resistant nanostructured surfaces are required to improve the wear and fatigue resistance of moving components.

In 2011, a large research project led by the Center members in IMR, Shenyang on synthesis and performance of nanostructured metals has been granted by the Ministry of Science and Technology of China under the program of National Key Basic Scientific Research Plan. Within the 5-year project with several Chinese research institutions involved, synthesis and performance of nanostructured metals will be further explored, targeting on development of high performance nanometals for industrial applications based on deeper understanding of the structure-property relationships of metals on the nanometer scale. This new project complements very well the work planned for a next 3 year Center period.

International collaboration outside the Center has also been intense covering partners all over the world. Concerning the Far East, two new projects have been initiated with the Sino-Danish Centre for Education and Research. Both are related to the use of nanometals in future fusion reactors. One is on oxide dispersion strengthened (ODS) steels and the other on nanostructured tungsten. Both projects involve Risø DTU and Tsinghua University and include also IMR Shenyang (ODS steels) and the Institute of Plasma Physics, Hefei (nanostructured tungsten). Two PhD students have been hired in 2011 for the projects. Also in the Far East, extensive collaboration with Japan on understanding and practical applications of nanometals is ongoing and has been intensified. This includes a project on 'Bulk nanostructured metals' with Kyoto University, and a project on 'Void formation and fracture of dual phase steel' supported by Nippon Steel, with the objective of optimizing microstructure and properties for a wide range of applications. The latter company is financing a PhD student inscribed at DTU and a new joint project on 3DXRD characterization of Goss grains in silicon steel.

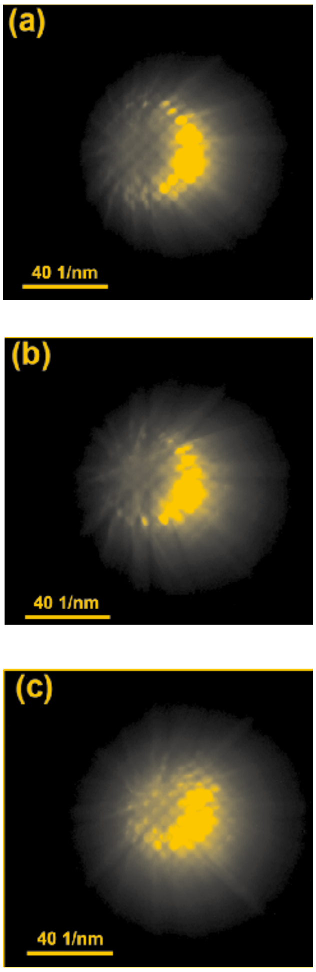
Internationally, the focus on materials is rising. On June 24th, 2011, President Obama launched the so-called "Materials Genome" programme with the goal of reducing the cost and time of new materials to market, based on integrated computational materials engineering (ICME). Three general focal areas were defined for ICME, namely materials development, process modeling and materials behavior. It is expected that this large American initiative will spread to the rest of the world and it is encouraging that the three focal areas match the work in our Center.

As to the future of the Danish partner, the DTU top management has decided that Risø National Laboratory for Sustainable Energy will cease to exist. Instead the laboratory will be converted into two large DTU institutes and one nuclear center located at the Risø site – to be called the Risø Campus. The two new institutes will be devoted to Wind Energy and to Energy Conversion and Storage. In practice this means that contradictory to the recommendations of an international evaluation held in September 2011, the Materials Research Division will be split into three major parts. The Danish part of our Center will, together with a large part of the Division from January 1st 2012, belong to the new Wind Energy Institute, which then will have a significant activity on Materials Science and Engineering. Under the given conditions this is considered to be the optimal solution, as the new institute will follow the Risø tradition of combining basic and applied research. It is also important that currently installed wind turbines need significant improvements of the metal parts to extend their lifetime, and our research on light and strong nanometals including hard wear resistant surfaces match these engineering challenges very well. Three of the present Center Risø scientists have chosen to join other DTU institutes. This will not affect the work within the present Center period much, as they will finish on-going research projects in 2012. Organizational wise, however, the present Danish daily coordinator senior scientist, Grethe Winther, will be replaced by scientist Chuanshi Hong from January 1st, 2012.

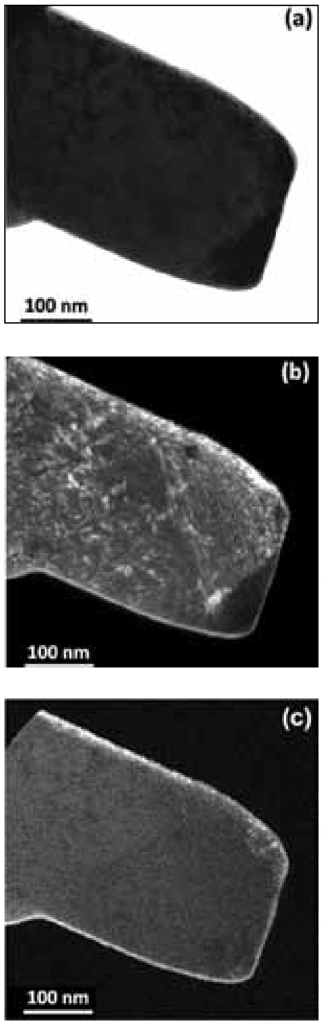
Also about the future, just before Christmas we received the good news that our application for a three-year extension of the Center was approved, with a significant increase in the funding of the Chinese partners. We are ready, enthusiastic and well prepared for the new Center period. The Danish-Chinese collaboration is working well - three Chinese PhD students, who have been "Danish Post Docs" at Risø, have been appointed to the scientist positions - and the new extended project plan is ready. Moreover, an international scene is set which matches our goals, and new key international collaborations are already in place. These include collaboration with Leuven University, Belgium and Sandia National Laboratory, USA on MD and phase field simulations of nanostructures, as well as with Cambridge University on plastic deformation of metals and with Manchester University, UK on advanced characterization on hard metals. Finally the next Risø International Symposium on Materials Science has been announced for September 3-7, 2012, with the theme "Nanometals – Status and Perspectives", where the "Center-family" will meet together with leading international scientists in our field. We hope you enjoy reading the highlights from 2011 and share with us our excitement for the Center in 2012 and the years beyond that.

Plastic deformation of submicron-size crystals followed by in-situ transmission electron microscopy (TEM)

The plastic deformation of submicron-size copper single crystals in the form of pillars has been characterized during in-situ compression in a transmission electron microscope using a state-of-the-art holder (PI-95 PicoIndenter) in a collaboration between Risø DTU and Tsinghua University. The variation in the local orientation during deformation has been followed by in-situ convergent beam electron Kikuchi diffraction (CBEKD). The in-situ observations have been followed by post-deformation measurements with the samples still mounted in the electron microscope. Crystal breakup following localized deformation has been observed in two of three crystals examined, and for all crystals the direction of rotation during deformation is in agreement with slip taking place on a subset of the four slip systems with the highest Schmid factors on the (111) and (-1-11) slip planes. A diffraction-based Burgers vector analysis confirms that the active dislocations observed by TEM are from slip systems with the highest Schmid factors. These results from testing of micropillars are in good agreement with the deformation behaviour previously reported for both single- and polycrystal samples with dimensions in the millimetre range. The pillar dimensions are 500×250×200 nm^3 and the experiment therefore demonstrate a bridging of length scale of deformation mechanisms.



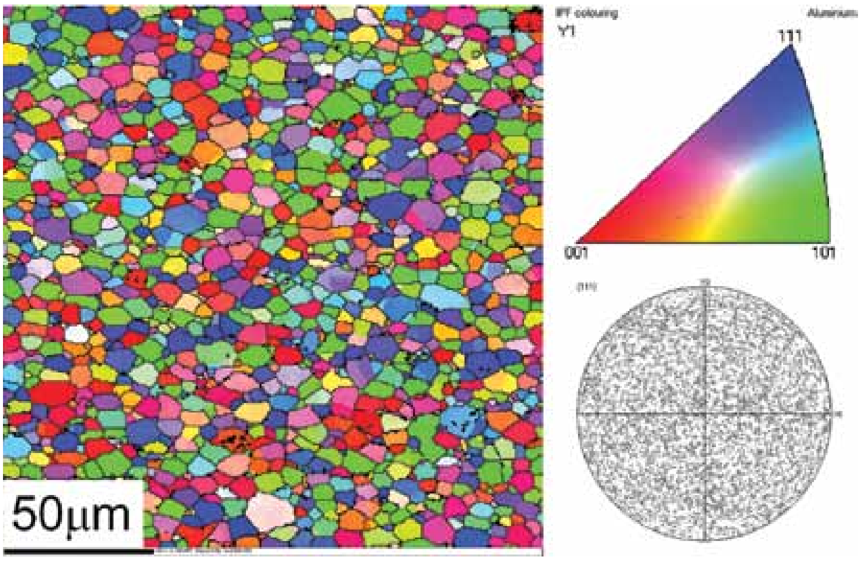
Kikuchi patterns taken from the in-situ measurement of crystal rotations of the area positioned at the half-height of pillar 1 at the 0th (a), 50th (b) and 80th (c) second, corresponding to strains of 0, 17% and 27% of the pillar.



Bright field (a) and dark field TEM images of pillar 3 after deformation to a strain of about 33% using g(200) (b) and g(020) (c), showing a fairly random distribution of dislocations and the formation of one dense dislocation wall

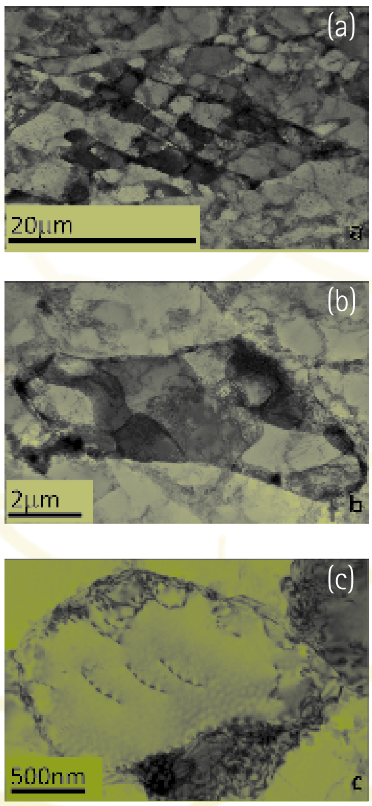
Polycrystals with submicrometer grain sizes

To investigate the grain size dependence of dislocation boundary formation during plastic deformation, samples have been prepared of aluminum with a range of ultrafine grain sizes. Previous similar studies have been hampered by the fact that top-down processed samples with an ultrafine grain size still contain a significant fraction of low angle dislocation boundaries. Instead therefore the Al samples were produced by powder compaction using the spark plasma sintering (SPS) process. By controlling the powder properties and the SPS conditions fully recrystallized material with a low dislocation density was achieved containing grains of size down to less than 0.1 µm.



Microstructure in SPS sample of Al with a 5µm grain size showing grains with a nearly random distribution of orientations

The as-prepared samples were deformed by compression testing to strains of up to 0.3. In the largest grains examination of thin foils in the TEM revealed that planar-type dislocation boundaries were formed, following a similar orientation dependence to that seen in conventional coarse grain material. With decreasing grain size transitions were seen first to dislocation boundary cell (non-planar) structures, and then to loose tangles. In the finest grains only loose tangles or isolated dislocations were found. The differences in dislocation structure could be correlated with a change in the work hardening during compression. In addition tensile testing on miniaturized samples was carried out, revealing the samples to have excellent ductility (up to 25% in the 5µm average grain size sample).

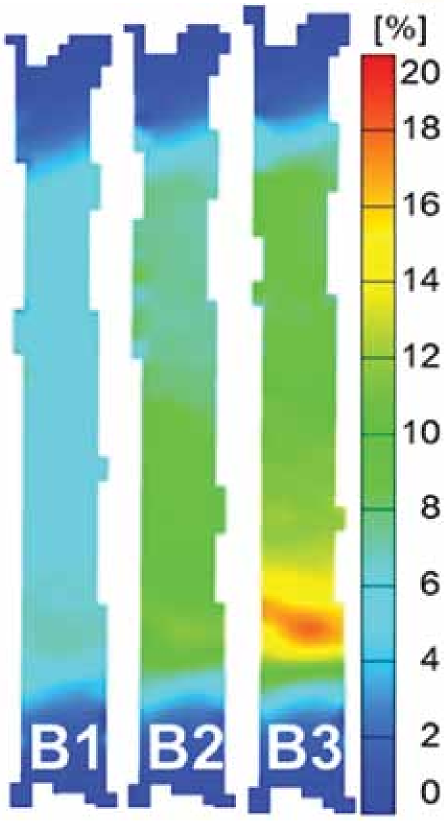


TEM images showing effect of decreasing grain size on dislocation storage and boundary formation during compression of Al SPS samples; (a) large grain showing extended planar boundaries; (b) typical grain in 5 µm average grain size SPS sample showing equiaxed cells; (c) typical grain in 0.7 µm average grain size SPS sample showing loose dislocations only.

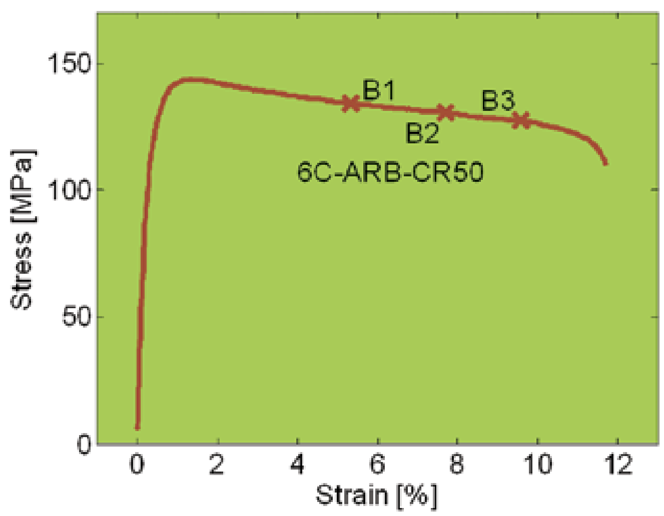
Investigation of necking behavior of aluminum

A nanometal produced by plastic deformation has a very high strength but a low ductility as the stress during loading is located in a small volume. By developing a new process it has been possible to increase the ductility significantly of aluminium illustrated by the stress-strain curve.

To explore the underlying mechanisms the strain distribution during loading has been analyzed in a collaboration between Risø DTU and IMR by using a system, ARAMIS, at IMR. In this test the specimen is covered with a pattern of dots allowing the strain distribution in the whole specimen to be followed during a tensile test. Results are illustrated here to the right for 3 different strains B1, B2 and B3 marked in the figures. In the figure to the right the different colors show the magnitude and distribution of strain illustrating the increasing strain localization when the strain is raised from B1 to B3. The results show that the material deforms uniformly up to a strain of about 6%. This indicates that the new way of processing aluminum leads to a promising combination of very high strength and formability. The test results are used to analyze and model the deformation behavior of the material and also to develop forming techniques for the practical applications of the new material.



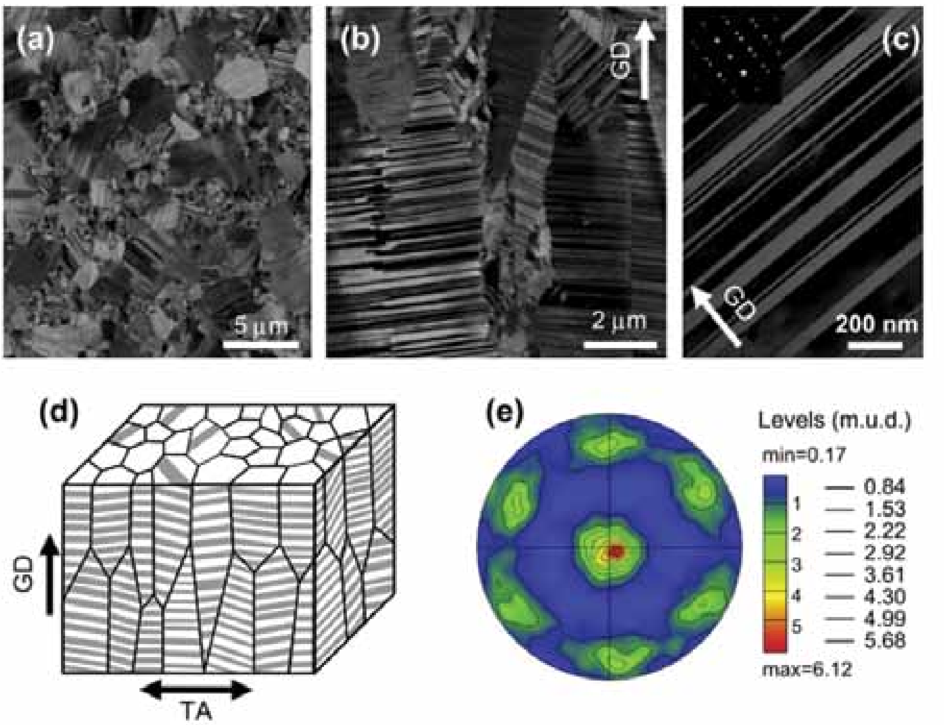
Strain distributions from a specimen with a width of 5 mm and gauge length of 32 mm. The colors show the magnitude of the strain in accordance with the scale bar. B1 to B3 represent specific tensile strain locations marked in the figure below



Stress-strain curve for an aluminium deformed to high strain by accumulative roll bonding and deformed 50% by cold rolling.

Tensile behavior of columnar grained Cu with preferentially oriented nanoscale twins

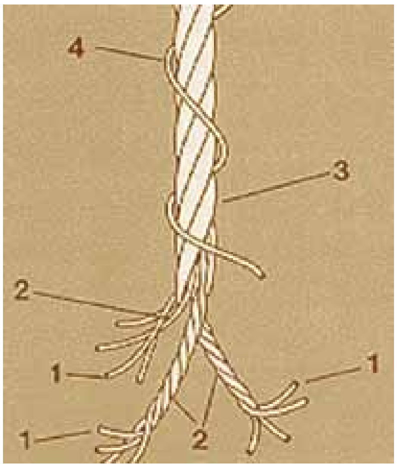
The mechanical properties of nanotwinned (nt) Cu is intimately correlated to the grain boundary and twin boundary (TB) morphology, grain size and twin thickness etc. We have synthesized a new kind of nanotwinned microstructure with columnar grains that grow along the deposition direction by direct current electrodeposition. Inside each grain, there are high density of nanoscale twins, most of which are parallel to the deposition plane. Uniaxial tensile tests show that the mechanical behavior of columnar grained nt-Cu is strongly dependent on the grain size and twin thickness. The yield strength is still dominated by twin thickness, thus reduction in twin thickness results in an increase in yield strength, but the increment is smaller than that expected from a Hall-Petch relationship. This deviation is related to a different deformation mechanism between dislocation and TBs. In the model of Hall-Petch strengthening, dislocations effectively pile up against TBs and GBs which directly obstruct the motion of dislocations. However in the columnar grained nt-Cu, dislocations no longer pile up on TBs since the tensile direction is parallel to TBs; they propagate along the channels between neighboring TBs instead. This dislocation behavior resembles the confined layer slip model frequently applied in the analysis of conventional nanoscale thin films or multilayer materials, where TBs play a role in confining dislocation slip. On the other hand, larger plastic deformation was found to occur in the vicinity of GBs and to propagate towards grain interiors. The localized GB deformation leads to high-density dislocation debris accumulated along GBs, which gradually evolve into dislocation cells and subgrains upon further straining. In some regions with larger strain, destruction of growth TBs was also observed. Obviously, the concentrated GB deformation intensifies the dynamic recovery of dislocations, affecting both work hardening and tensile ductility. Since the volume fraction of GB affected areas decreases with increasing grain size, the uniform tensile ductility is expected to strongly depend on grain size, which is supported by the observation of a significant increase in ductility when the grain size exceeds 3 μm. This work reveals a new deformation mechanism of nt-metals with preferentially oriented twin structures and further advances the understanding of strengthening and deformation mechanism of a nanotwinned structure.



SEM-BSE images of the as-deposited Cu sample in (a) plane view and (b) cross-section view. (c) TEM cross-section observation. (d) 3D illustration of the overall microstructure of as-deposited Cu. (e) pole figure showing the strong {111} out-of-plane texture.

Steel with super strength

In a collaboration between Tsinghua University and Risø DTU, the density and distribution of dislocations in drawn steel wires have been investigated by TEM and High Resolution Electron Microscopy (HREM). It is found that the characteristics of the dislocation structures change significantly as the structure refines with increasing strain and the strength increases. These microstructural observations are used in a theoretical analysis of dislocation generation, storage and annihilation which can underpin the strength-structure relationship observed experimentally.



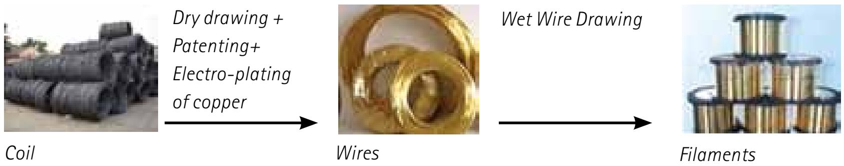
1. Filament

2. Strand

3. Cord

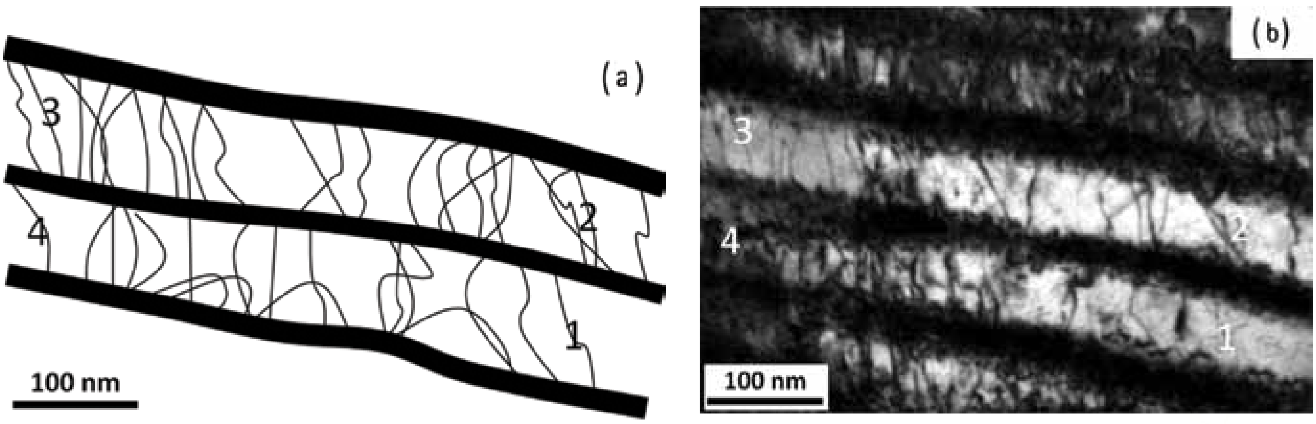
4. Spiral

Production prosess of filaments



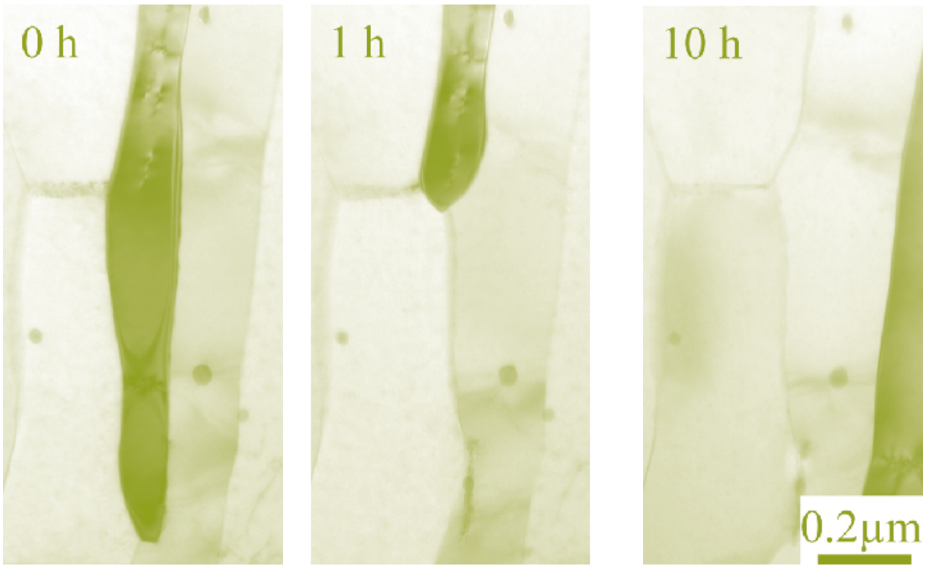
Steel cords for automobile tires

(a) A sketch and (b) a TEM micrograph of the structure in a sample deformed to a strain of 0.68, showing dislocations in the ferrite lamellae.

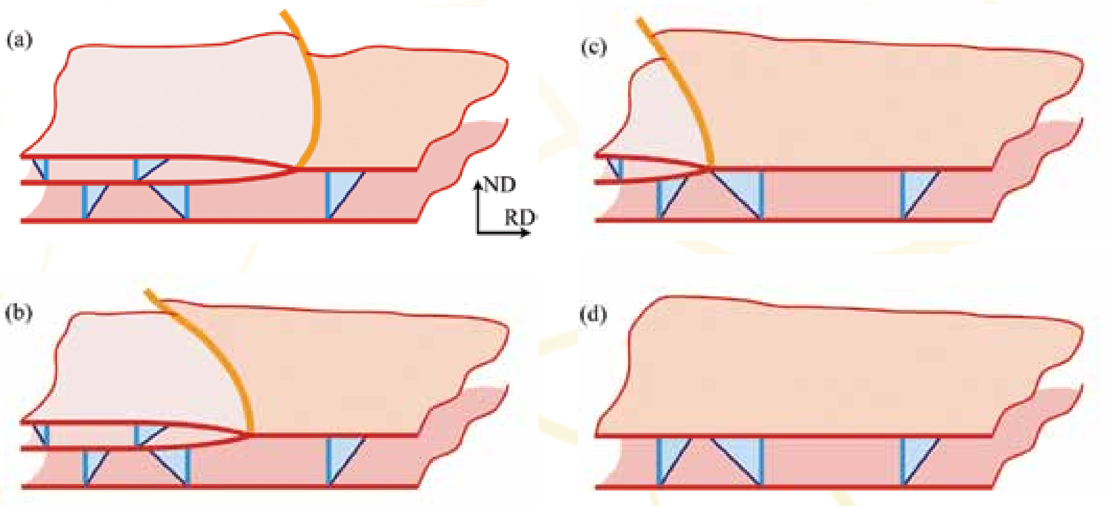


Recovery by triple junction motion in nanostructured aluminum

Nanostructural aluminum produced by deformation to ultrahigh strains has excellent strength properties but reduced thermal stability. Recovery and recrystallization can therefore take place even at low temperatures and the material will lose strength. Therefore in order to find measures to stabilize the microstructure recovery mechanisms at low temperatures has been studied by transmission electron microscopy (TEM) and electron backscattered diffraction (EBSD). Experimental conditions were optimized and a new mechanism was discovered – recovery by triple junction motion. By this mechanism deformation induced dislocation boundaries and grain boundaries can move and annihilate at low temperatures, the structure will coarsen and the strength will decrease. It has also been observed that the boundary movement can be blocked by small particles which points to means by which the structure and mechanical properties of nanometals can be stabilized thereby enhancing their applicability in society.



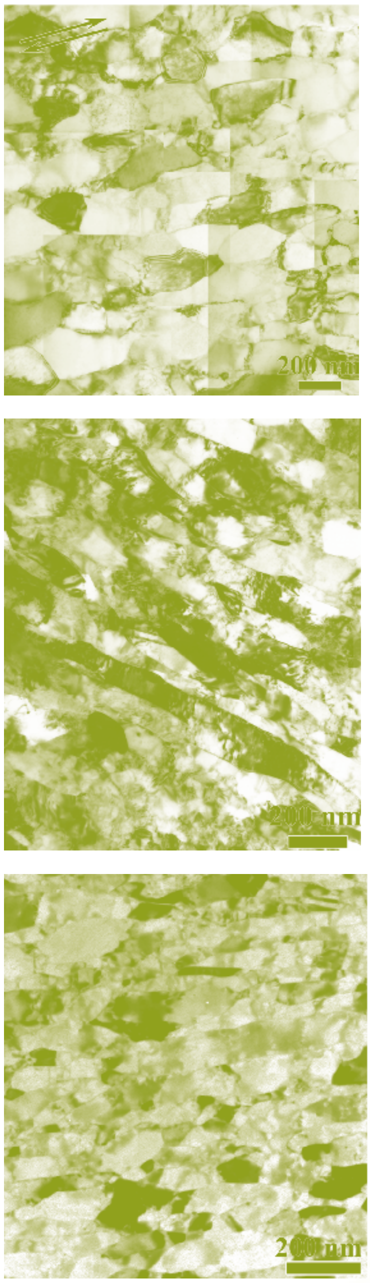
TEM observations at the same area of heavily deformed Al between annealing steps, showing triple junction motion, which leads to removal of the dark lamella in the middle and consequent widening of neighbouring lamellae.



A schematic illustration of triple junction motion in a finely spaced lamellar structure. The migrating triple junction is shown in amber, and its migration leads to removal of the confined lamella and consequent widening of neighbouring lamellae.

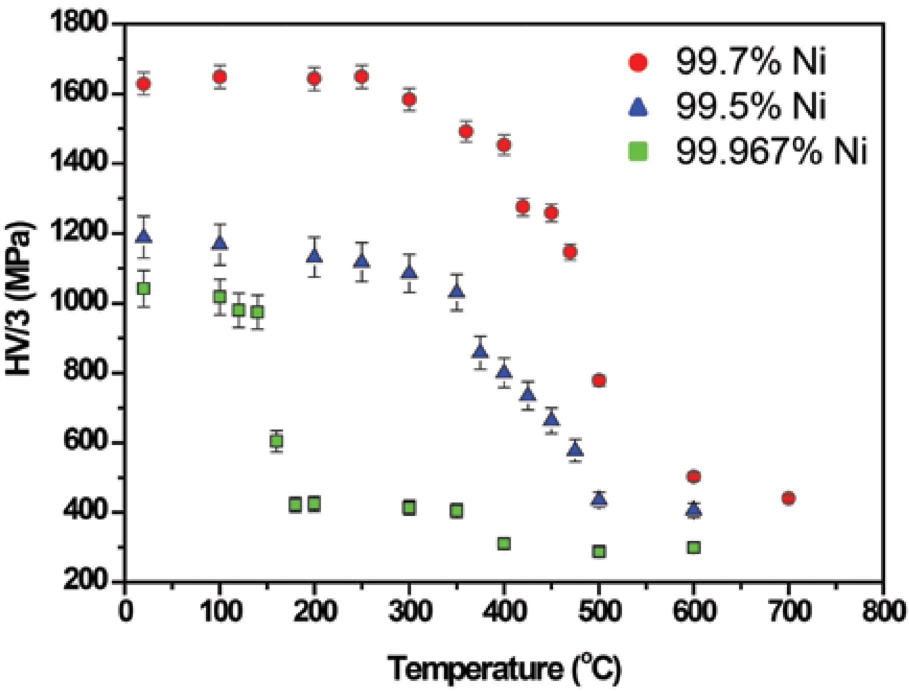
Thermal stability and finer structures through light alloying

In the development of nanometals for engineering applications, it is important to improve their thermal stability. The effect of adding small amounts of alloying elements has been explored in a cooperation between IMR SYNL, Risø DTU and Erich Schmid Institute in Leoben (Austria). It has unexpectedly been found that even small additions of alloying elements down to 0.1-0.4 wt.% can significantly improve the properties of nanostructured Ni produced by high pressure torsion (HPT). Alloying leads to delayed recovery and recrystallization kinetics and a large increase in the temperature interval over which these processes take place. At the same time the spacing between the deformation-induced boundaries is reduced significantly from about 130 nanometers in pure nickel to below 50 nanometers and the result is a significant increase in strength. These findings open a window of using slight alloying to produce tailored nanometals with optimized mechanical properties and thermal stability.



Structure of nickel deformed by HPT to a strain of 100. The boundary spacing in 99.967% pure (top) is bigger than in 99.5% pure nickel (middle) and 99.7% pure nickel (bottom).

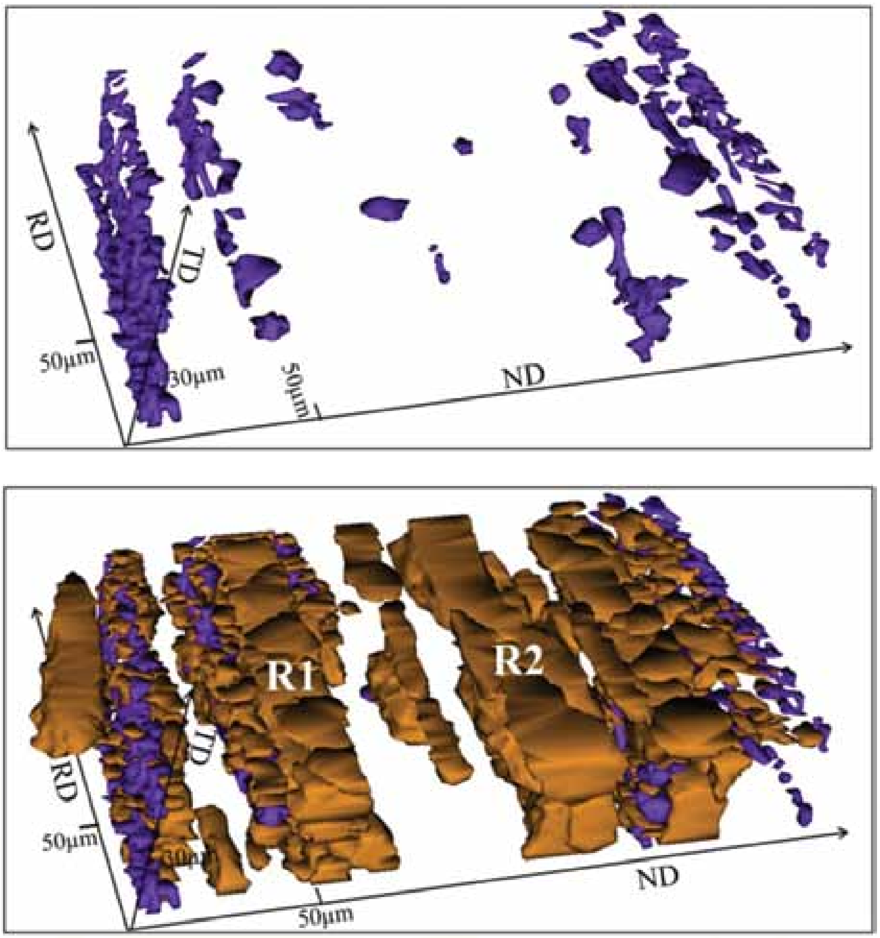
The mechanical strength (hardness on the vertical axis) increases with alloying and is maintained at high temperatures.



3D investigation of recrystallization nucleation in a particle-containing Al alloy

In a collaboration between Chongqing University and Risø DTU serial sectioning was used to study effects of an inhomogeneous distribution of second-phase particles on the nucleation of recrystallization in aluminium (AA 3104). 3D maps of the particles and the nuclei were reconstructed based on 2D electron channeling contrast micrographs and electron back scattering patterns. In total 2423 nuclei were identified which are about 10 times more nuclei than has previously been characterized in 3D. The present 3D investigation confirms the results of earlier 2D investigations, namely that large particles in clusters are very powerful nucleation sites. In the present sample they stimulated the formation of 90 % of the observed nuclei.

Investigation further showed that the size of the nuclei varies significantly and relates to the matrix in which they form and grow – for example nuclei in zones with rod shaped dispersiods were surprisingly observed to be larger than those in zones without particles. The result is explained by the heterogeneous spatial distribution of the various types of particles in the present material. This is important as the inhomogeneous distribution of constituent and dispersoid particles due to micro-segregation during ingot casting is a key problem for successful modeling of subsequent hot deformation and recrystallization in sheet metal producing industries.



3D reconstruction of big intermetallic particles in the whole inspect volume (top), showing that most of the particles align in bands or clusters when seen from the RD-ND plane, and (bottom) 3D reconstruction of both big intermetallic particles and nuclei (totally 368 nuclei), showing that most of the nuclei are next to big particles and align in bands or clusters, too. Nuclei in regions R1 and R2 are obvious larger than in other regions.

Exchange

Visits from China to Risø DTU, Denmark

Prof. Andy Godfrey, Tsinghua
Prof. Lei Lu, IMR SYNL
Prof. Nairong Tao, IMR SYNL
PhD student Guomin Le, Tsinghua
PhD student Hui Tian, Beijing Uni. Tech.

Visits from Risø DTU to Tsinghua University

Dr. Techn. Niels Hansen
Dr. Techn. Dorte Juul Jensen

Visits from Risø DTU to IMR SYNL:

Senior Scientist Xiaoxu Huang
Senior Scientist Wolfgang Pantleon
PhD student Jacob Kidmose

Visits from Risø DTU to Chongqing University

Dr. Techn. Niels Hansen
Dr. Techn. Dorte Juul Jensen
Senior Scientist Xiaoxu Huang
Senior Scientist Wolfgang Pantleon

Publications

Journal Articles

1. Chen X.H., Lu, L., Lu, K., Grain size dependence of tensile properties in ultrafine-grained Cu with nanoscales twins, Scripta Materialia, 64(2011)311.

2. Fang, T.H., Li, W.L., Tao, N.R., Lu, K., Revealing extraordinary intrinsic tensile plasticity in gradient nano-grained copper, Science, 331(2011)1587.

3. Hansen, N.; Juul Jensen, D., Deformed metals – structure, recrystallization and strength, Materials Science and Technology, 27 (2011) p. 1229-1240.

4. Huang, F., Tao, N.R., Lu, K., Effects of impurity on microstructure and hardness in pure Al subjected to dynamic plastic deformation at cryogenic temperature, Journal of Materials Science & Technology, 27(2011)628.

5. Le, G. M.; Godfrey, A.; Hong, C. S.; Huang, X.; Winther, G., Orientation dependence of the deformation microstructure in compressed aluminum, Scripta Materialia 66 (2012) p. 359–362.

6. Liu, H. H.; Schmidt, S.; Poulsen, H. F.; Godfrey, A.; Liu, Z. Q.; Sharon, J. A.; Huang, X., Three-Dimensional Orientation Mapping in the Transmission Electron Microscope, Science, 332 (2011) p. 833-834

7. Luan, B. F.; Hansen, N.; Godfrey, A.; Wu, G. H.; Liu, Q., High-strength Al-Al2O3p composites: Optimization of extrusion parameters, Materials and Design, 32 (2011) p. 3810-3817.

8. Luo, Z. P.; Mishin, O. V.; Zhang, Y. B.; Zhang, H. W.; Lu, K., Microstructural characterization of nickel subjected to dynamic plastic deformation, Scripta Materialia, 66 (2012) p. 335-338

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Public Outreach

An official Chinese news agency, Xinhua News, made a video interview about the Danish-Chinese Center for Nanometals, which was distributed in China. In the video, senior scientists Xiaoxu Huang and Grethe Winther explained what nanometals are, why nanometals represent an important research field with a great potential benefit for individuals and society, what achievements have been obtained in the center, and how the strong collaboration between Danish and Chinese scientists have made these achievements possible.

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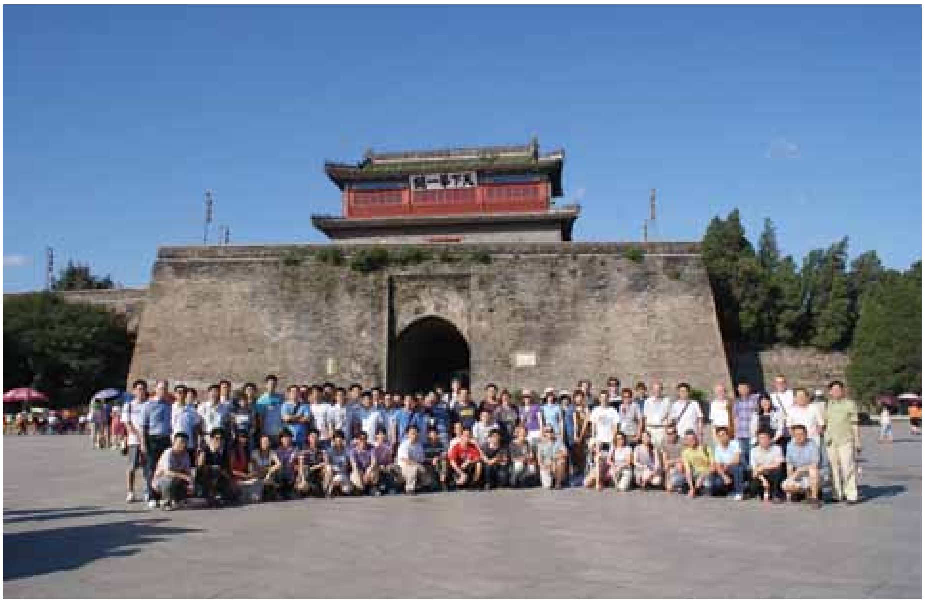

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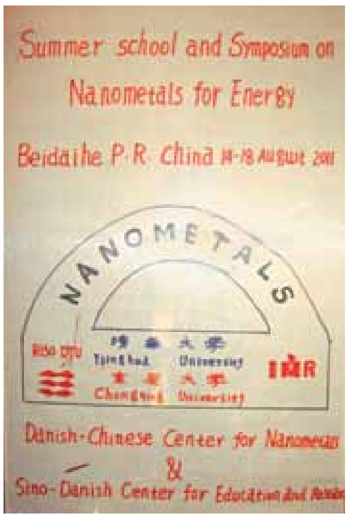

Conference Proceedings

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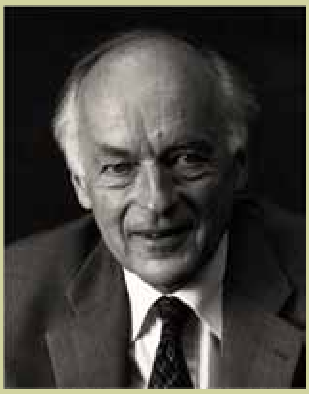
Honors and Awards



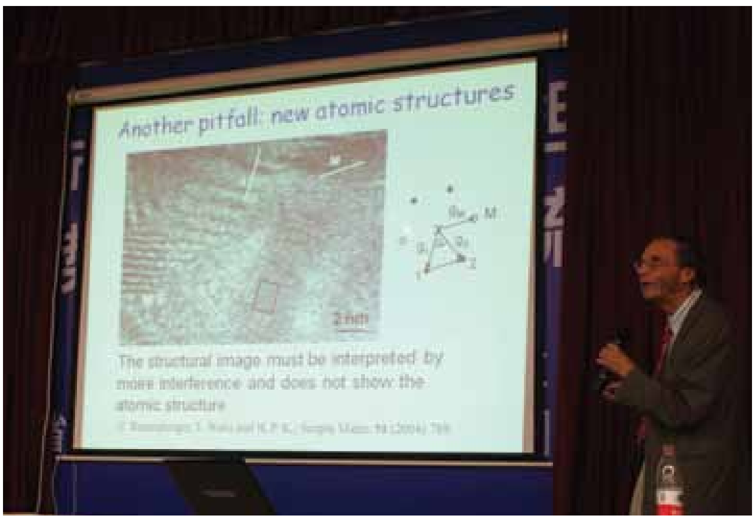
Dr. Techn. Niels Hansen was awarded the Einstein Professorship by the Chinese Academy of Sciences.

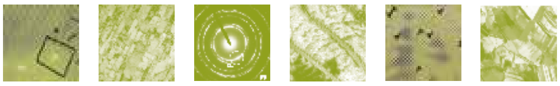
Dr. Techn. Dorte Juul Jensen was awarded the Danish Knighthood.

Prof. Ke Lu received the Humboldt Research Award.

Other

1. Huang, X., Chapter 10, “Characteristic structures and properties of nano-structured metals prepared by plastic deformation” in: Nanostructured metals and alloys, edited by Sung H. Whang, Woodhead Publishing Limited, Cambridge (2011) p. 276-295.





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