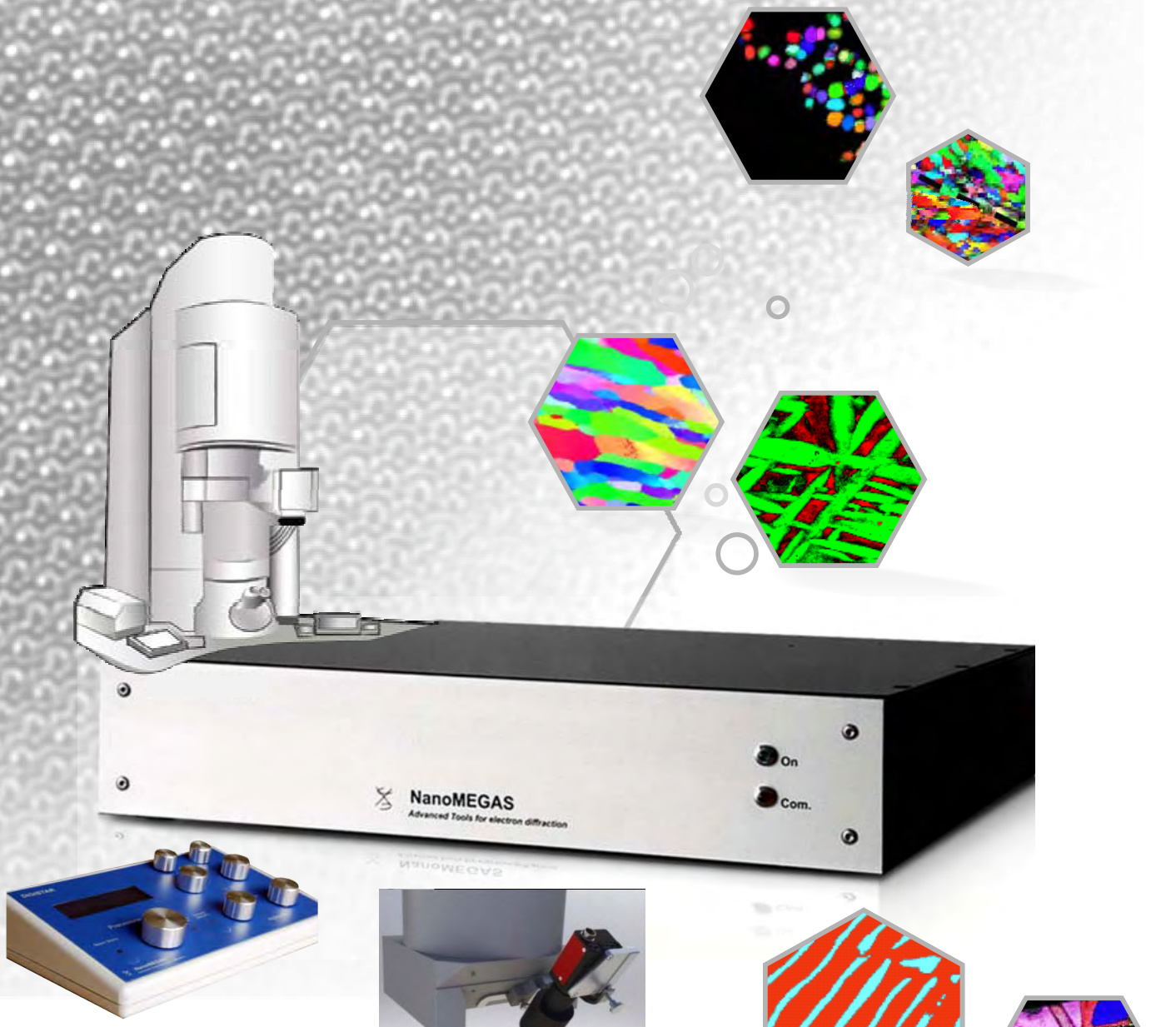
ASTAR

Application Notes
Nanoscale TEM Orientation Imaging Analysis



NanoMEGAS
Advanced Tools for electron diffraction

Nanoscale TEM Orientation and

ASTAR

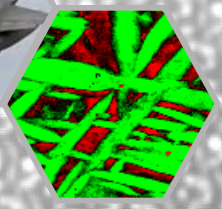
Ceramics

Metals

Nanowires

Polymers

Metals



Spintronics

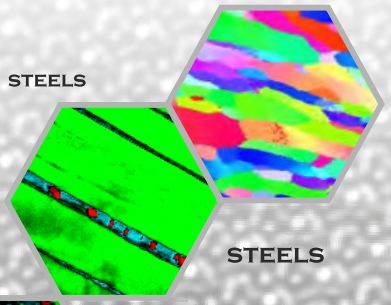
Nanomagnets

Batteries

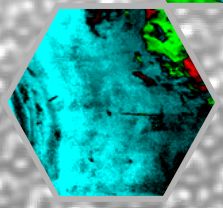
Metals

NanoMEGAS
Advanced Tools for electron diffraction

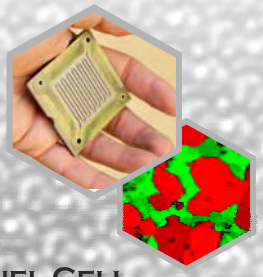
Phase Imaging Analysis



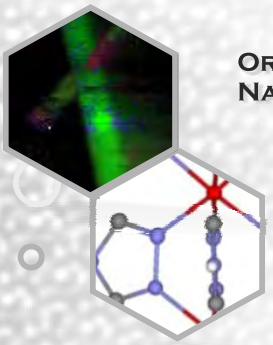
Steels



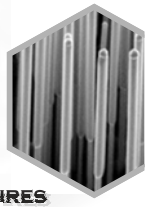
Steels



Fuel Cell



Organic Nanomagnets

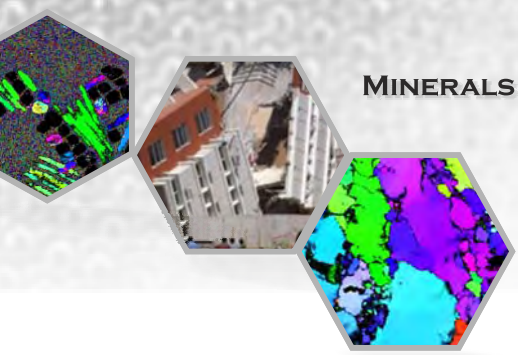


Nanowires

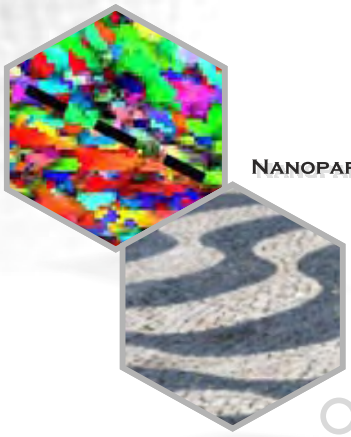
Materials science properties (metals, alloys, Ceramics, polymers, semiconductors,nanoparticles) depend on great extent on their textures at nm scale level . ASTAR device uses TEM based orientation mapping technique (EBSD-TEM like) based on collection of precession elecron diffraction (PED) patterns and cross-correlation comparison with simulated templates.

ASTAR can turn any TEM into a very powerful analytical tool enabling orientation-phase imaging at 1 nm resolution in combination with other analytical techniques

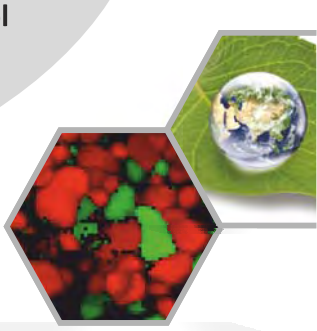
Nanoparticles



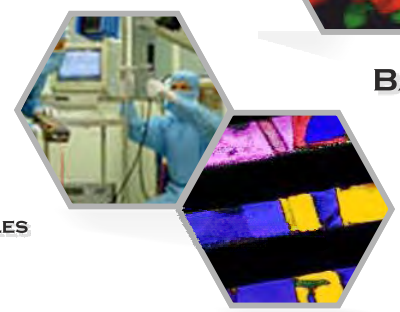
Minerals



Nanoparticles



Batteries



Semiconductors

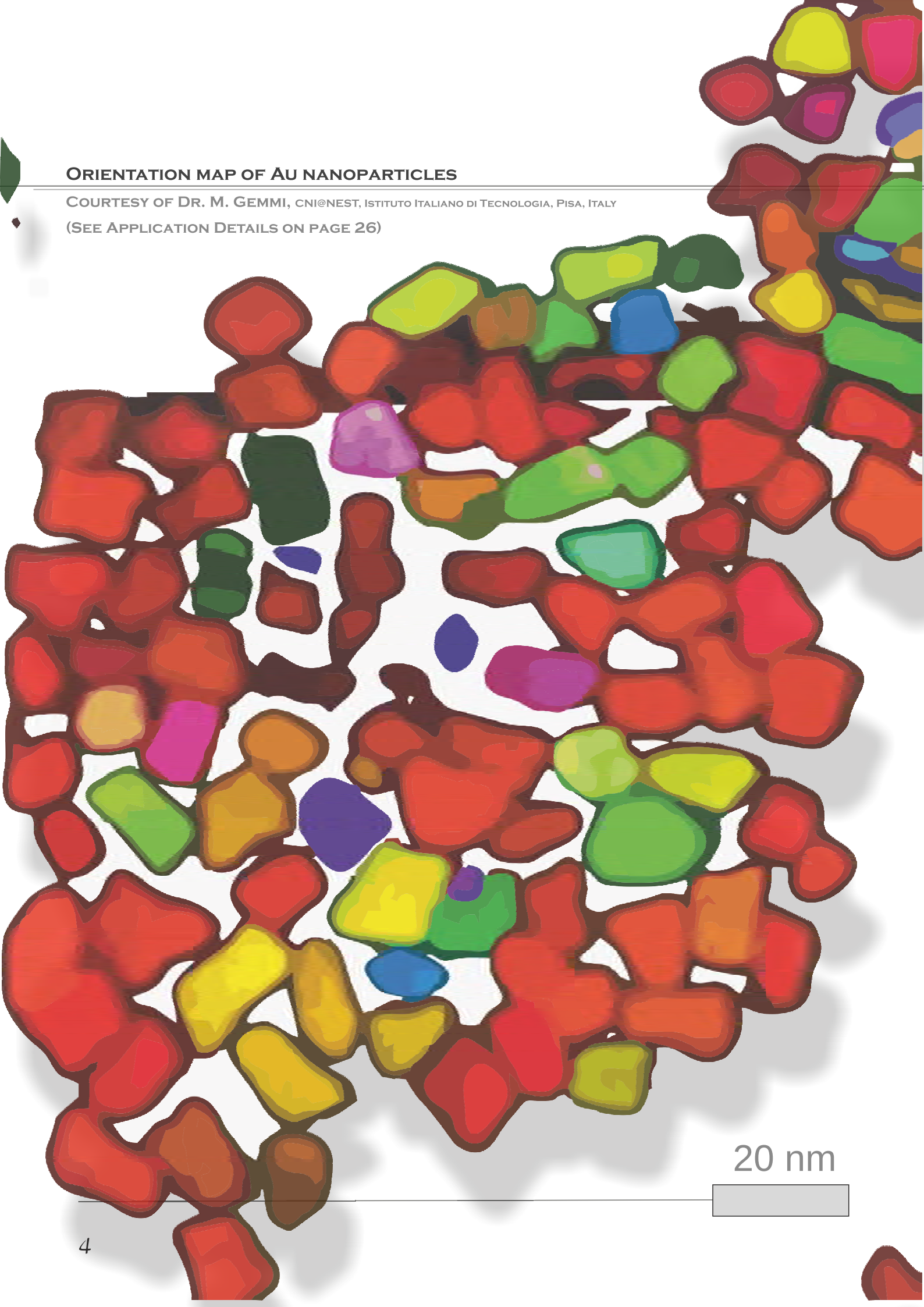
Polymers

Metals

Orientation map of Au nanoparticles

Courtesy of Dr. M. Gemmi, cni@nest, Istituto Italiano di Tecnologia, Pisa, Italy

(See Application Details on page 26)

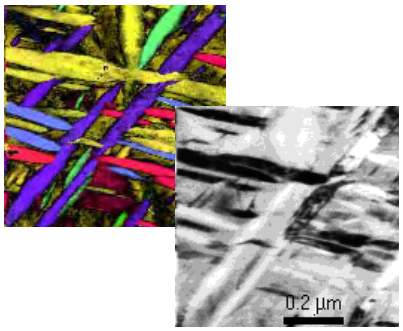


Challenge

Reveal Texture At Nanometer Scale

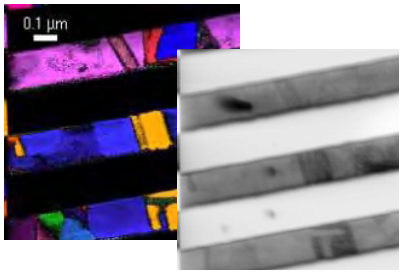
Metals / Alloys

Texture of metals is linked to specific physical properties, so the need to characterize it at nanometer scale



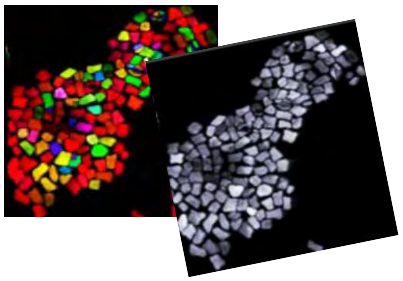
Semiconductors

Faster chip performances in electronic devices push Copper Interconnects in close to nanometer scale



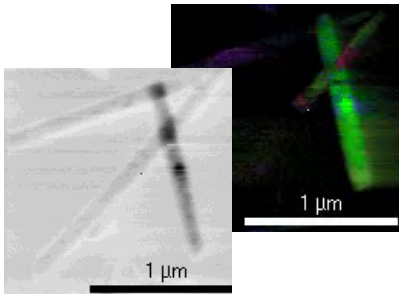
Nanoparticles

Nanoparticle size and texture are very important for drug delivery of poor water soluble drugs



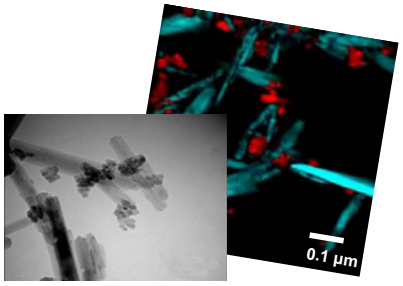
Polymers /organics

Crysralline polymers and other organics (pharmaceuticals) need new techniques for structure characterization



Minerals

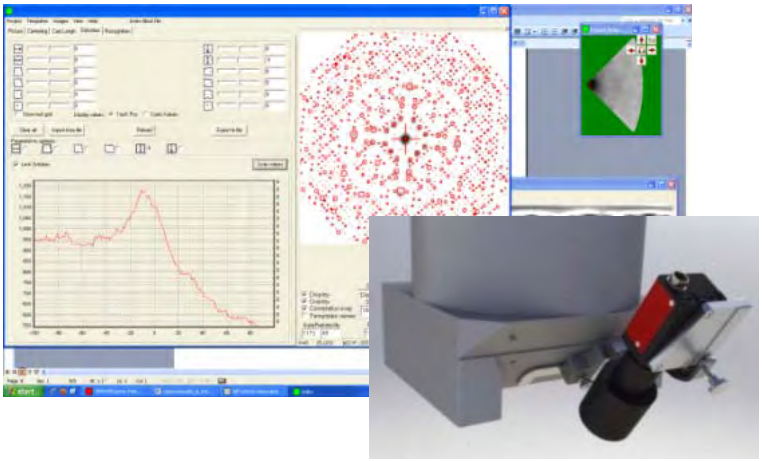
Textures of minerals at nm scale are intimately linked to their physical & chemical properties

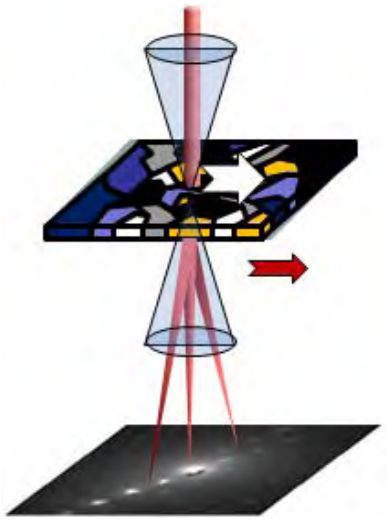


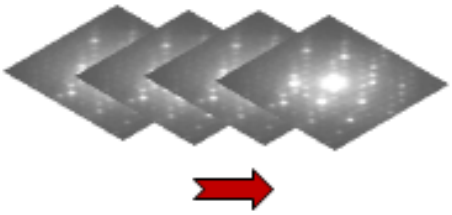
SOLUTION

AUTOMATIC TEM ORIENTATION/PHASE MAPPING PRE

HOW IT WORKS



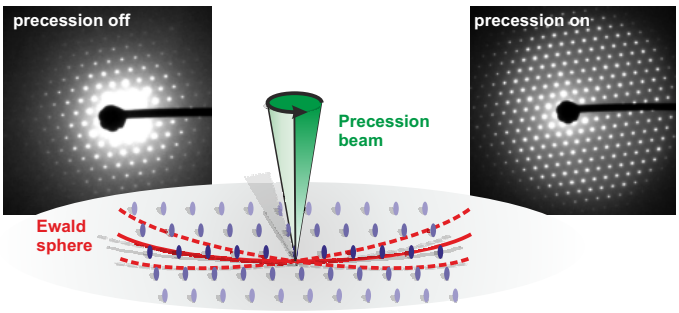




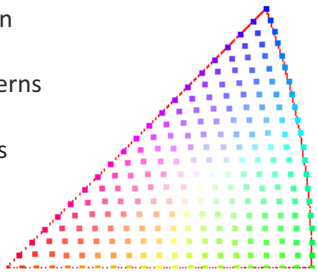
ASTAR is an automatic crystallographic indexing and orientation/phase mapping tool, developed for any TEM.

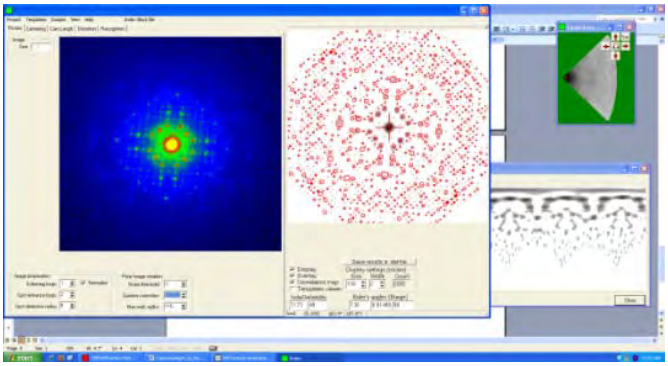
The electron beam is scanned in combination with beam precession through the sample area of interest (a); beam scanning is done by the NanoMEGAS DigiSTAR precession unit without using an inbuilt STEM mode.

A number of electron diffraction (ED) spot patterns from several sample locations (b) are acquired at high speed using a dedicated fast CCD camera (c) placed in front of the TEM screen; local crystal orientation(s) are obtained by comparing all individually obtained ED spot patterns via cross-correlation matching techniques with pre-calculated ED templates.

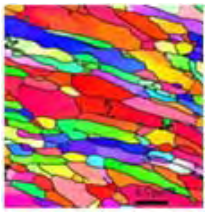


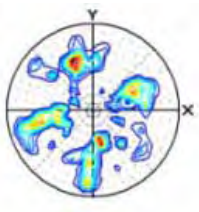
Detection and orientation/phase mapping of different (known) crystallographic phases and orientations in a crystal structure requires collection of high quality ED patterns. Electron beam Precession diffraction is extremely useful for obtaining patterns with a large number of spots (d) almost twice as many compared to conventional (e)

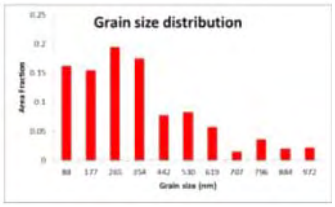




HOW IT WORKS







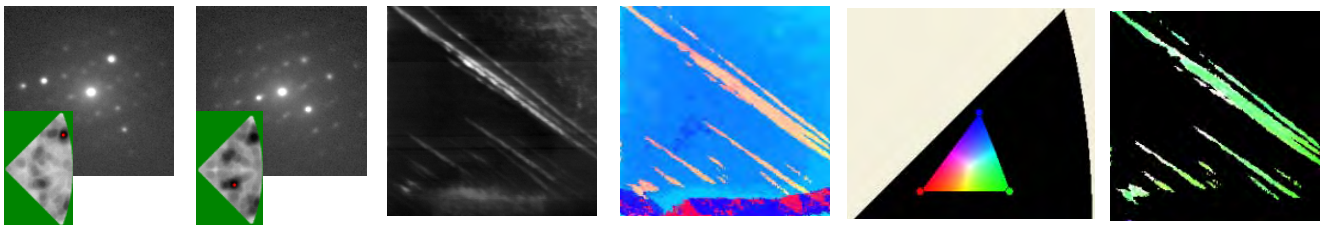
selected area electron diffraction (SAED) and without dynamical effects (e.g. Kikuchi lines). Therefore, phase and orientation identification based on pattern matching between experimental precession patterns with simulated templates is very reliable and precise. Required diffraction templates are generated every 1º through the respective symmetry invariant section (f) of orientation space (stereographic triangle for cubic crystals). Resulting colour maps show with nm detail grain structure including boundaries (g), grain size distribution, and relative pole figures.

ASTAR resolution on a TEM is being determined by the electron probe size and can reach 1 nm on orientation maps with TEM-FEG microscopes.

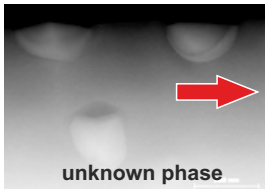
Virtual Stem (BF/DF)

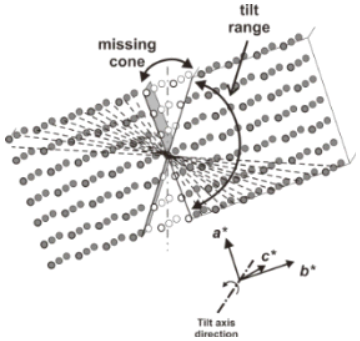
ASTAR orientation-phase maps leads to **digital files** where information (e.g. about the corresponding ED pattern) can be retrieved from every pixel. It is possible then to create virtual bright / dark field Iimages by selecting specific reflections from the ED pattern. In the example below (**TWIP deformed steel**), a number of twins are present that can be revealed either using a **virtual aperture centered on twin reflections** (b-c)

(a) ED pattern corresponding to matrix reflections, (b) ED pattern with twin reflections and virtual aperture centered on a particular reflection, (c) corresponding virtual dark field showing twins, (d) orientation map, (e) pole figure with color code corresponding to twin orientations within the stereographic triangle (f), Courtesy Prof. S. Godet, Univ. Brussels (ULB), Belgium.

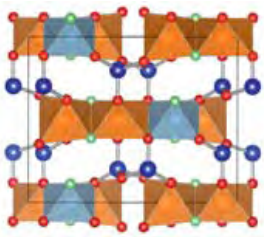


Solving Unknown Structures : 3D Precession Diffraction Tomography





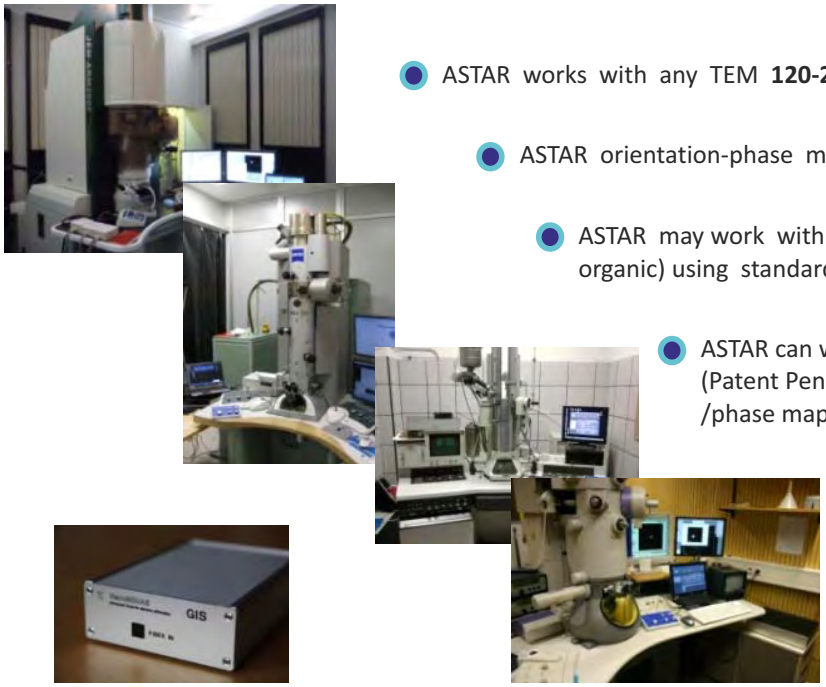
Courtesy Ute Kolb group, Mainz



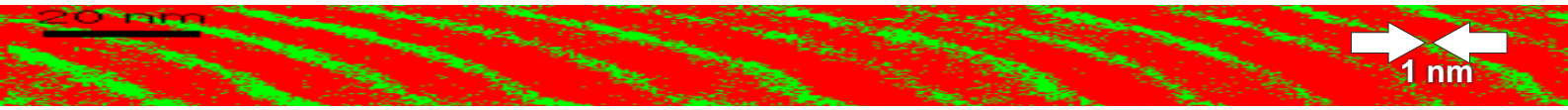
TEM allows to study nm size crystals and by tilting (manually / automatically) around an arbitrary axis a single nanocrystal (tilt usually > 120 ° e.g 120 ED parterns with step 1 °) in combination with precession diffraction (usually 1 °), the reciprocal cell of any unknown phase can be recontructed and crystal cell can be evaluated automatically (error 2-5%). ED intensities can also be measured automatically to enable complete solution of crystal structure (optional software).

Specifications

TEM Automated Orientation / Phase Imaging Analysis



- ASTAR works with any TEM **120-200-300 Kv** (**LaB6 /W- FEG**)
- ASTAR orientation-phase map **1 nm resolution** with TEM-FEG *
- ASTAR may work with any type of diffracting material (inorganic / organic) using standard TEM specimen preparation techniques
- ASTAR can work in combination with beam precession (Patent Pending technique) for ultra-precise orientation /phase maps and ab-initio solution of unknown phases
- ASTAR can work / retrofit between multiple TEMs in same lab
- Galvanic Isolation system (GIS) via optical fiber for ASTAR-TEM connection

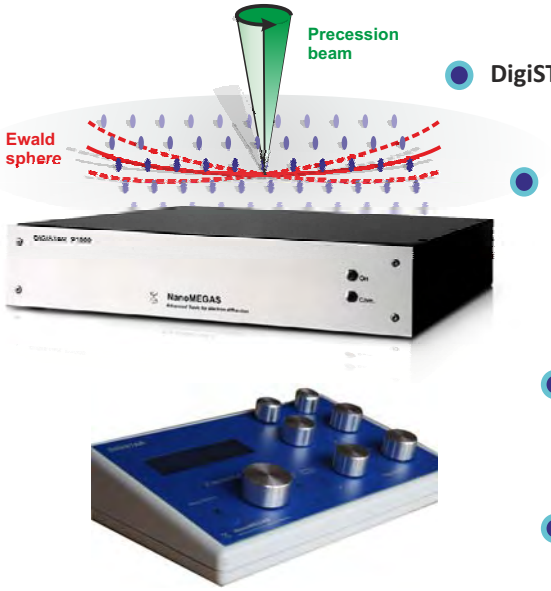


ASTAR includes

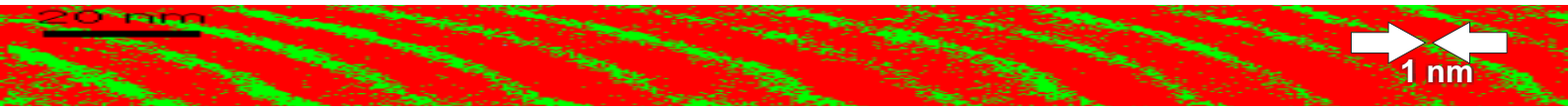
- Digital Scan Generator for area beam scanning – no need for STEM unit
- Scanning step from 0.1 nm to 100 nm in TEM / nanobeam /microprobe mode (TEM dependent)
- ASTAR dedicated highly flexible ultra-fast (> 100 frames/s; 256 gray levels) CCD Stingray camera to capture ED patterns to produce high speed orientation maps / diffraction pattern calibrations
- CCD Stingray can be placed in front of TEM fluorescent screen via special mechanical adaptor

- ASTAR/ CCD Stingray camera can be adapted / retrofitted to any TEM in less than 5 minutes
- ASTAR CCD Stingray can be perfectly stay together with any other on axis / 35 mm port CCD cameras / GIF system

ASTAR INCLUDES



- **DigiSTAR digital precession unit compatible with any TEM**
- Beam precession 0-4° & frequency 0.1-2 kHz
  (values TEM configuration dependent).
  Precession angle 0.5-1.0° recommended for ASTAR orientation imaging
- **DigiSTAR –TEM alignment values are memory stored**
- **Manual user interface** for precession angle visualization and precise alignment adjustments



ASTAR (SOFTWARE)

- **TEMDPA: Transmission Electron Microscope (TEM) Diffraction Pattern Acquisition**
  Computer system workstation generates software that controls signal over different channels connected to TEM deflecting coils. Stingray CCD camera can be used for data (ED patterns) acquisition.

- **DIFFGEN : Diffraction pattern generator**
  ED simulated templates are generated every 1° (or less) through the Ewald for every phase / crystal symmetry
  *** User friendly graphical interface (diffraction pattern, pole figure)**
  *** Diffraction patterns generated for all crystal systems.**

- **INDEX : Orientation identification through Diffraction Pattern Matching**
  Every calculated-simulated pattern generated with DIFFGEN is compared to the experimental measured one through template matching
  *Optimizing routines for orientation resolution <1°; automatic camera length calibration
  *Correlation Index map calculation and display (for reliability checking)
  *Pseudo-bright field or pseudo dark field image reconstruction

- **MAP VIEWER : Orientation map viewing software**
  Crystal orientations extracted for every pattern related to the scanned area are stored in result files.
  • **Orientation map : pixel color is related to the sample x, y or z crystallographic direction.**
  * **Phase map : pixel color is related to different existing crystal phases** (eg cubic, hex, tetragonal) in crystals
  Crystallographic features : grain boundaries may be apparent on orientation maps, grain size and pole figures analysis.
  • Export facilities compatible with the most available image / EBSD-SEM analysis software (TSL, HKL etc)

* 1 nm resolution tested on reference Al 9nm / 1 nm TiN nanolayer composite sample (see application note) and 0.4 nm thick twin structure detected at < 5 nm Pd nanoparticles