

DIGISTAR

Advanced TEM electron diffraction tools for
nanocrystal structure determination



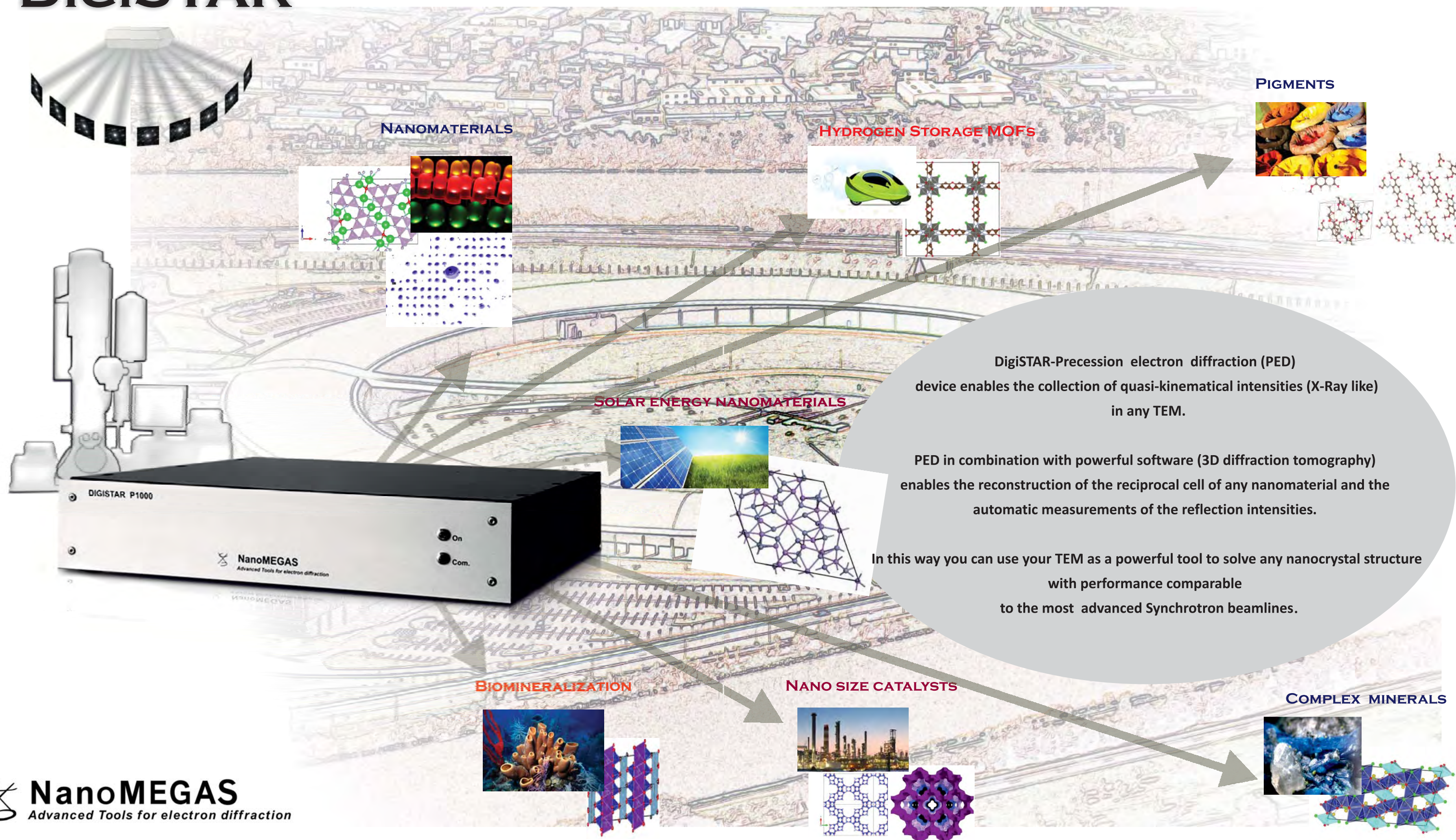
APPLICATION NOTES 3D PRECESSION DIFFRACTION TOMOGRAPHY



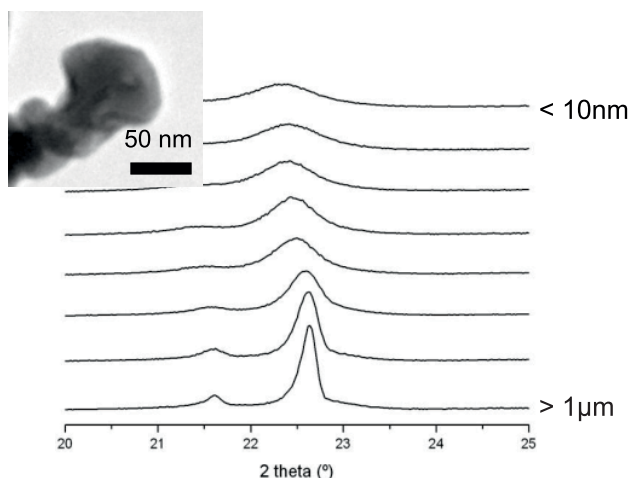
ENABLE SYNCHROTRON PERFORMANCE WITH YOUR TEM

3D NANOCRYSTAL STRUCTURE DETERMINATION

DIGISTAR



CHALLENGE SOLVING ANY NANOMATERIAL STRUCTURE

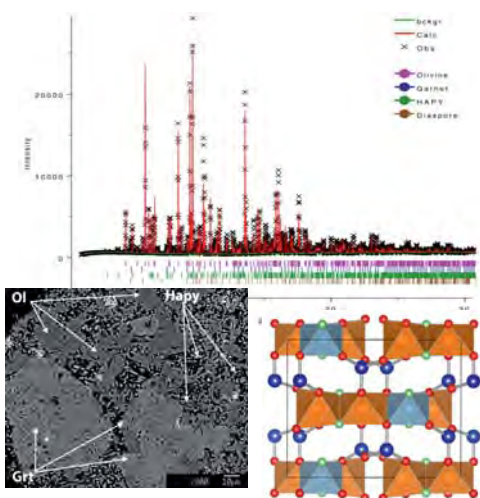
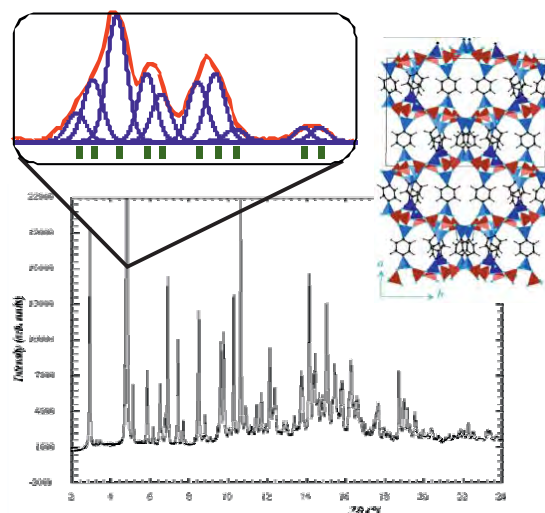


SMALL CRYSTALS

In case crystals are smaller than 0.5 micron in size, X-Ray reflections peaks are getting progressively broader thus making impossible the refinement of the crystal cell parameter.

TOO MANY OVERLAPPING X-RAY PEAKS

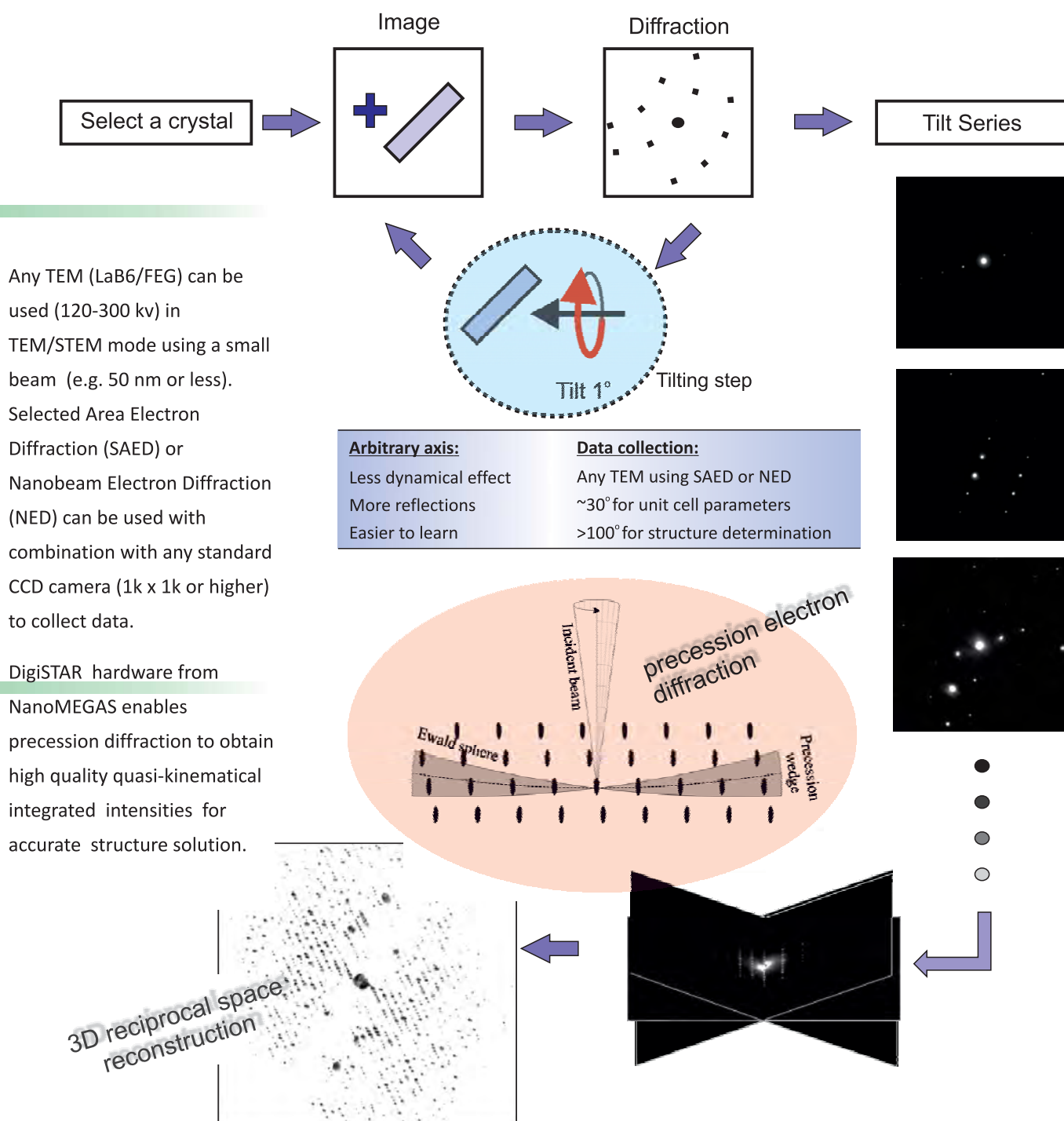
In case of poorly crystallized materials like pharmaceuticals, zeolites and inorganic-organic hybrid ECS-23 catalyst, Synchrotron X-Ray diffraction pattern is poorly defined with many overlapping peaks. Evaluation of crystal cell parameters and correct intensity measurement is impossible.



MANY CRYSTAL PHASES

In case of complex multiphase systems where new unknown phases are found in very small amounts (like the HAPY-Hydrous Al bearing pyroxene is synthesized as high-pressure phase in $\text{MgOAl}_2\text{O}_3\text{-SiO}_2\text{-H}_2\text{O}$ system), their presence cannot be detected in Synchrotron powder X-Ray pattern.

Transmission Electron Microscope (TEM) allows to study nm size crystals. By tilting (manually/automatically) around an arbitrary axis a single nanocrystal (tilt range usually $> 120^\circ$ e.g. 120 diffraction patterns with step 1°) in combination with precession electron diffraction (PED), the reciprocal cell can be reconstructed and crystal cell parameters can be evaluated automatically and precisely (error 2-5 %). Reflection intensities can also be measured automatically (completeness of reciprocal space $> 60\%$) to enable the solution of the crystal structure.



3D PRECESSION DIFFRACTION TOMOGRAPHY

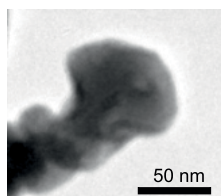
SPECIFICATIONS



Works with any TEM with 120-200-300 kV

LaB6 /W or FEG filament

Selected area (SAED) or Nanobeam (NBD) mode



Smallest crystal used: ~ 100 nm organics & ~ 20 nm inorganics

Application for agglomerated / embedded samples
Polyphasic materials



Diffraction data collection with **any CCD camera**
(35 mm port or on axis) 1k x 1k or higher

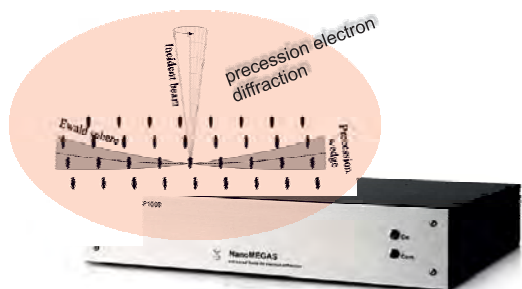
Diffraction data collection with single tilt holder / tomography holder
or cryoholder (for beam sensitive samples)

Minimum tilt (for cell parameters determination) $-15^{\circ}/+15^{\circ}$ 1° step
Recommended tilt (for crystal structure determination) $-45^{\circ}/+45^{\circ}$ 1° step



DigiSTAR compatible with any TEM

Beam precession ($0-4^{\circ}$) TEM configuration dependent
 1.2° recommended for PED tomography



Manual / automatic PED data collection
(TEM config. dependent)

Software for 3D diffraction tomography

Reconstruction of reciprocal space by collected PED patterns
Automatic cell parameter determination (2-5 % error)
Space group determination
Automatic measurement of 3D reflection intensities
for ab-initio structure determination of any structure

