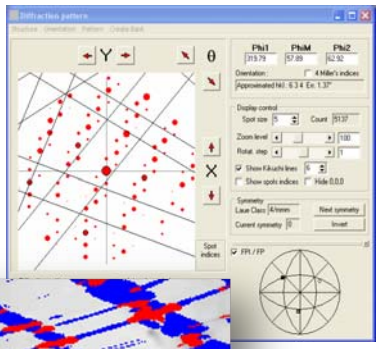
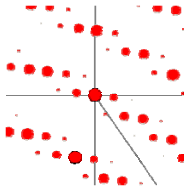
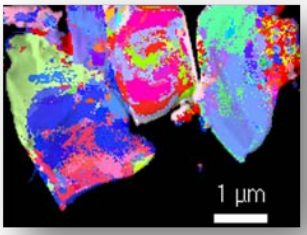
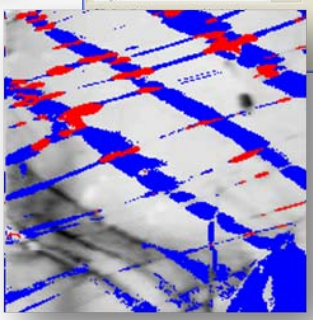
ASTAR

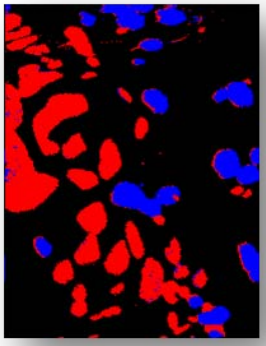
Automatic Orientation - phase mapping precession unit for TEM nanoanalysis











- ASTAR can easily be retrofitted to any (old or new) 100- 300 kV TEM
- TEM technique similar to EBSD - SEM (with much higher resolution details)
- Perform detailed nanocrystal orientation maps (Orientation resolution < 1º < 5 nm step size)
- Application for metals, ceramics , semiconductors and any type of diffracting material, any crystal symmmetry, no surface sample preparation
- Perform detailed crystal phase maps (resolution < 5 nm -TEM FEG < 25 nm - LaB6 TEM)
- Ultra-fast data adquisition (< 5 min) (eg 5x 5 microns, 500 x 500 points) with external CCD camera
- Sample scanning combined with precession for accurate orientation -phase maps *

* Patent pending

ASTAR : (EBSD-TEM) like solution for your TEM microscope

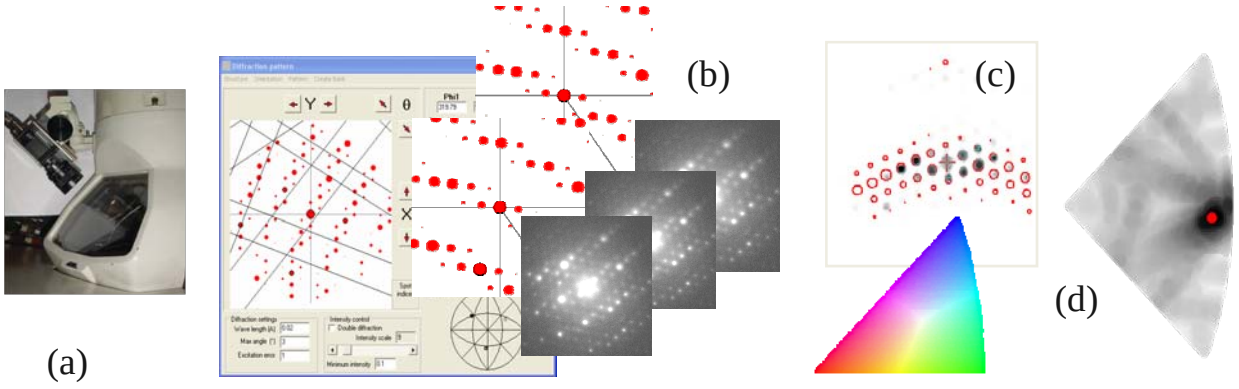


FIG.1 (a) CCD camera attached to TEM screen , automatic generation of ED templates (b) comparison with experimental ED patterns (c) via cross-correlation (d) until best orientation is found at the correlation index map .

ASTAR package is an automatic crystallographic orientation and indexing tool developed for any TEM. Electron beam is scanned through the sample surface and a big number of ED patterns from several locations are acquired thanks to a CCD camera ; local crystal orientation(s) are obtained by comparing individually all obtained spot ED patterns via cross-correlation techniques with pre-calculated ED templates (simulated for all possible crystal orientations); correct orientations are then revealed after comparing /selecting templates with experimental spot ED patters having highest correlation index.

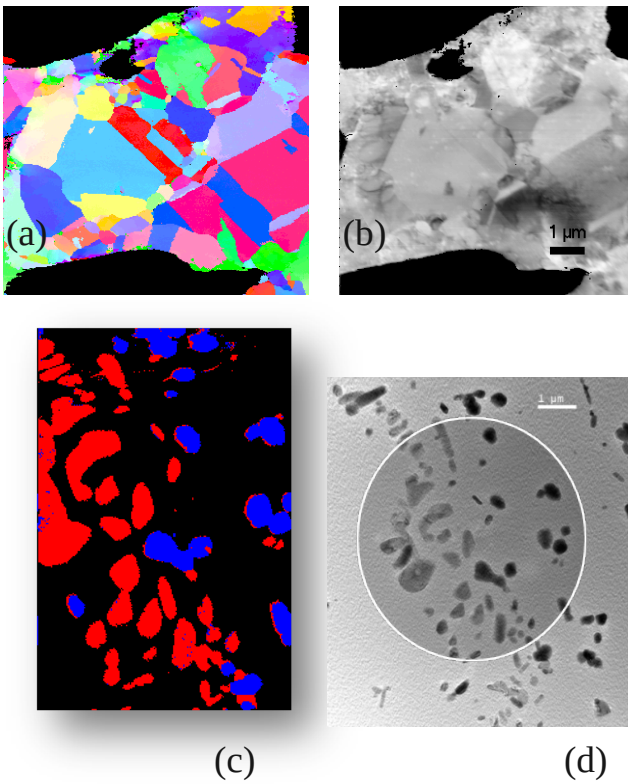


FIG.2 (a) **ASTAR** orientation map reveraling nanotwins in Cu sample (Jeol 3010 LaB6 , CBD mode ,12 nm resolution (b) pseudo-bright field area of the same sample (c) ASTAR phase map of circular area (d) of replica sample extracted from 430 stainless steel containing precipitates of cubic fcc $M_{23}C_6$ carbides and hexagonal Cr_2N nitrides (red stand for a = 0.483 nm and c = 0.451 nm nitride and blue stand for a = 1.062 nm carbide .

Courtesy Dr.J.Portillo,Univ.Barcelona,.Spain

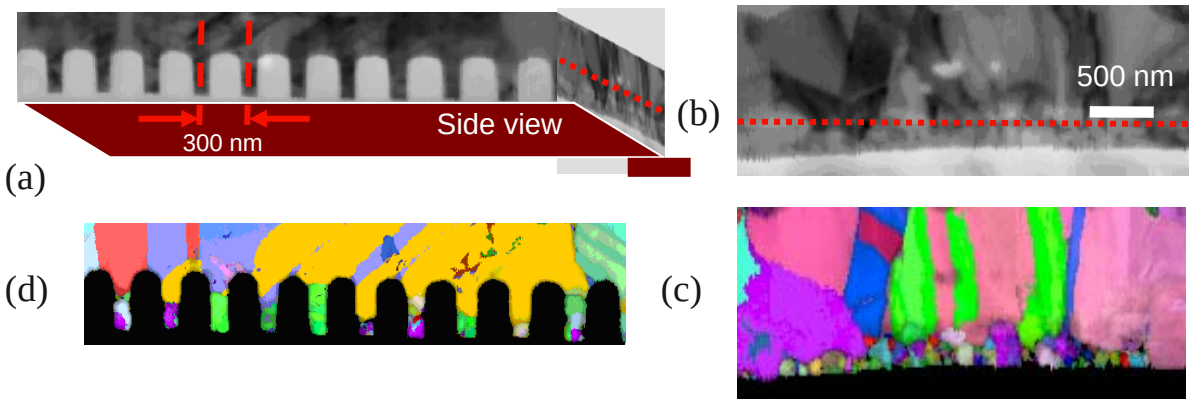
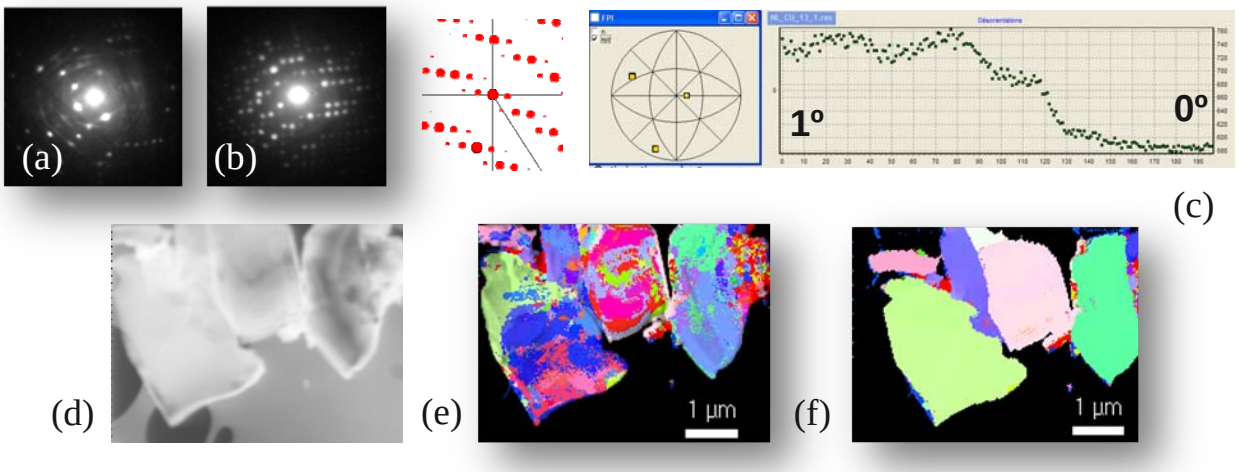


FIG.3 Semiconductor textures : ASTAR orientation texture map –side view (a) at 80 nm copper lines (Jeol 3010 LaB6 , 500x 100 steps , 6.5 nm each, spot size 25 nm), (b) (c) 250 x 100 steps 13 nm step spot size 25 nm (d) 300x 100 step , 6.5 nm each 15 nm spot size
Image courtesy S.Brandstetter , E.Rauch SiMap Grenoble France

ASTAR : (EBSD-TEM) precession and orientation maps in TEM

Detection and mapping of different crystallographic phases and orientations in a crystal structure requires collection of high quality ED patterns. **Electron beam Precession (1)** is extremely useful to obtain patterns with a big number of spots –almost twice compared with SAED- at large angles , therefore phase and orientation identification can be very reliable after comparison with simulated templates. Precession is possible with **dedicated unit (** "spinning star" P0x0 or "DigiSTAR" P1000) included in the ASTAR package and adaptable to any TEM (without need of STEM). Fig.4,5 show dramatic precision

FIG.4 Mayenite crystal $Ca_{12}Al_{14}O_{33}$ ED pattern without (a) and with (b) precession , (c) correlation index variation with increasing precession angle (from 0º to 1º) for fixed beam position and orientation. Orientation mapping of mayenite crystals (virtual bright field (d), without precession (e) and with precession angle 0.35º (f) Courtesy J.Portillo Univ.Barcelona Spain , E.Rauch SiMap Grenoble France



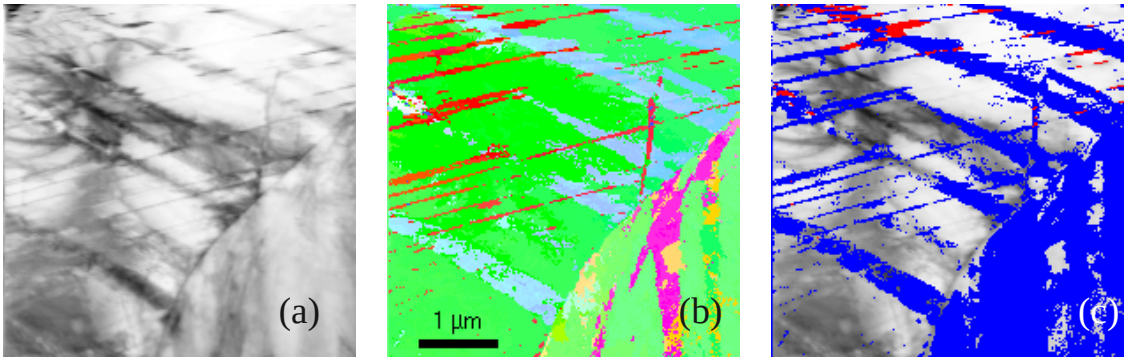
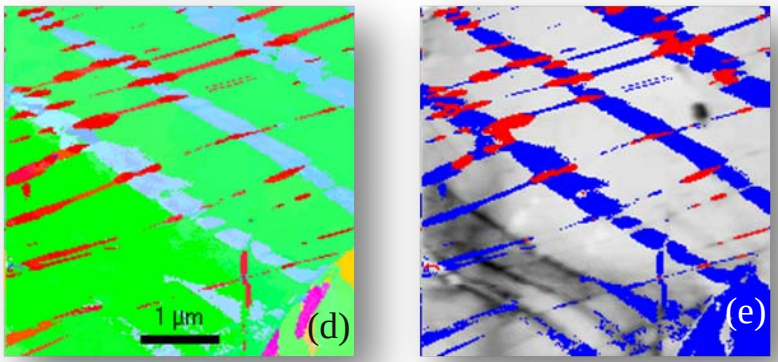


FIG.5 Austenitic stainless TRIP steel containing 3 disitint phases : γ fcc matrix, hcp ε bands and bcc α´ martensite at bands intersections (a) bright field TEM image coupled to orientation (b) ASTAR orientation and (c) phase map acquired without precession; same area orientation (d) and phase (e) map acquired with precession angle 0.4º (step size 22 nm). Courtesy E.Rauch, M.Veron SiMap Grenoble, France



improvement in both orientation and phase maps for many materials as precession eliminates orientation errors and ambiguities (like 180º ambiguity in cubic crystals). On the other hand precession reduces significantly dynamical diffraction effects in ED patterns and help solve crystal strutures ab-initio .

REFERENCES

1.Vincent R., P.Midgley Ultramicroscopy 53, 271-282 , 1994
2.E.F.Rauch ,M.Veron,J.Portillo,D.Bultreys,Y.Maniette and S.Nicolopoulos
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