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Electron microscopy / Microscopie électronique

Networking strategies of the microscopy community for improved utilization of advanced instruments: (2) The national network for transmission electron microscopy and atom probe studies in France (METSA)

Stratégies de mise en réseau élaborées par les communautés de la microscopie électronique pour optimiser l'utilisation des nouvelles plateformes instrumentales : (2) Le réseau français de microscopie électronique et de sonde atomique (METSA)

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A B S T R A C T

With the development, over the past ten years, of a new generation of electron microscopes with advanced performance, incorporating aberration correctors, monochromators, more sensitive detectors, and innovative specimen environments, quantitative measurements at the subnanometer and, in certain cases, at the unique atom level, are now accessible. However, an optimized use of these possibilities requires access to costly instruments and support by specialized trained experts. For these reasons, a national network (METSA) has been created in France with the support of CNRS and CEA in order to offer, in centres with complementary equipment and expertise, an open access to an enlarged and multidisciplinary community of academic and industrial users.

R É S U M É

Avec le développement, au cours de la décennie passée, d'une nouvelle génération de microscopes électroniques aux performances améliorées, équipés de correcteurs d'aberrations, de monochromateurs, de détecteurs plus sensibles ou d'une gamme innovante d'environnements autour de l'échantillon, les mesures quantitatives sont désormais réalisables à l'échelle sub-nanométrique, voire à celle de l'atome individuel. Cependant, l'utilisation optimale de ces possibilités requiert l'accès à des instruments coûteux et la participation d'un personnel expert dédié. Pour ces raisons, un réseau national (METSA) a été créé en France avec le soutien du CNRS et du CEA, pour offrir, dans des centres disposant de l'équipement adapté et d'un personnel entraîné, un accès ouvert à une large

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communauté interdisciplinaire d'utilisateurs en provenance du monde académique aussi bien qu'industriel.

Since the pioneering work of Ruska and Knoll [1] in the 1930's, Electron Microscopes (EMs) have regularly been improved: more stable electronics, from thermo-ionic to field-emission guns, faster and more sensitive detectors, etc. A major breakthrough appeared in the 1990's when Rose first proposed a design to build a spherical aberration corrector [2] that Haider built in 1995 with the CEOS Company [3]. This impressive development makes EMs unsurpassed tools for characterizing condensed matter, providing spectroscopic and structural quantitative information down to atomic level. Although these improvements lead permanently to better performance in terms of resolution, detection limits and sensitivity, ability to measure and not simply observe, EMs became more and more complex instruments and therefore more and more expensive. In addition, advanced EM techniques like: HR(S)TEM, EELS, Holography, cryo-microscopy, tomography etc. do require more and more expertise.

These reasons have been a significant driving force for the creation in France of a national network to facilitate access to advanced EM and to the required expertise in it, and therefore to promote advanced characterization of materials at the nanoscale by microscopy techniques. In addition, such a network should naturally increase the synergy and collaborations in the French community. According to the evident complementarities of Transmission Electron Microscopy (TEM) with Field Ion Microscopy (mainly its more recent developments as Atom Probes (AP)), these two techniques were associated in the project since the beginning. In January 2009, the METSA network (*Microscopie électronique en transmission et sonde atomique*, www.metsa.fr) was thus created by the CNRS and the CEA, with the support of the French Ministry for Research and Higher Education.

Although not restricted to it, METSA is mostly appropriate for Materials Science, with application from Physics, Nanotechnology to Engineering through Chemistry, Earth and Environment Sciences with interactions with Life Sciences. The network initially associated seven main microscopy centres or platforms, and it grew to eight centres (but ten laboratories) in 2013. The current METSA partners are listed in Table 1, together with their main expertise areas, which are further briefly illustrated in Fig. 1.

The status of METSA evolved in 2012 from an Emergent Program to a CNRS Research Federation (FR 3507) [4]).

Role and organisation of METSA

The main objectives of METSA can be summarized as follows:

– METSA aims at promoting the access to state-of-the-art and advanced means of TEM and AP by the French and international academic and industrial community, in the fields of condensed matter sciences (as indicated above), and in complement to the use of conventional laboratory tools.
– METSA provides a mean to support scientific developments, possibly in both instrumentation and methodology, with a link towards the international community.[1]
– METSA intends to play a role in the organisation of the French community in terms of teaching, training and advice if considered as useful by the governmental institutions, regarding the needs and prospects in the field of advanced microscopy.

Table 1
Members of the METSA Research Federation FR CNRS 3507.

Platforms	Location	Expertise
CEMES, UPR 8011	Toulouse	C_s-corrected HRTEM, EELS, in situ, holography
IM2NP[a], UMR 6242	Marseilles	Atom Probe (Tomography), C_s-corrected HRTEM
CiNaM[a], UMR 3118	Marseilles	Environmental TEM
MATEIS[b]-CLYM, UMR 5510	Lyons	All purposes TEM, Environmental EM
PFNC-Minatec (CEA)	Grenoble	C_s-corrected HRTEM – HRSTEM, EELS
LPS, UMR 8502	Orsay	C_s-corrected HRSTEM, STEM-EELS
MPQ, UMR 7162	Paris	C_s-corrected HRTEM, EELS
CRISMAT[c], UMR 6508	Caen	Quantitative Electron Diffraction, heating/cooling
GPM[c], UMR 6634	Rouen	Atom Probe (Tomography), Field Ion Microscopy
IPCMS, UMR 7504	Strasbourg	TEM tomography, EFTEM, C_s-corrected HRSTEM

[a] Partners of the CIME-PACA platform.
[b] MATEIS laboratory is the main of the 13 partners of the FED CLYM.
[c] Members of the IRMA CNRS Federation (FR 3095).

[1] 2 METSA platforms are members of the ESTEEM/ESTEEM2 project (see the specific contribution in the present special issue).

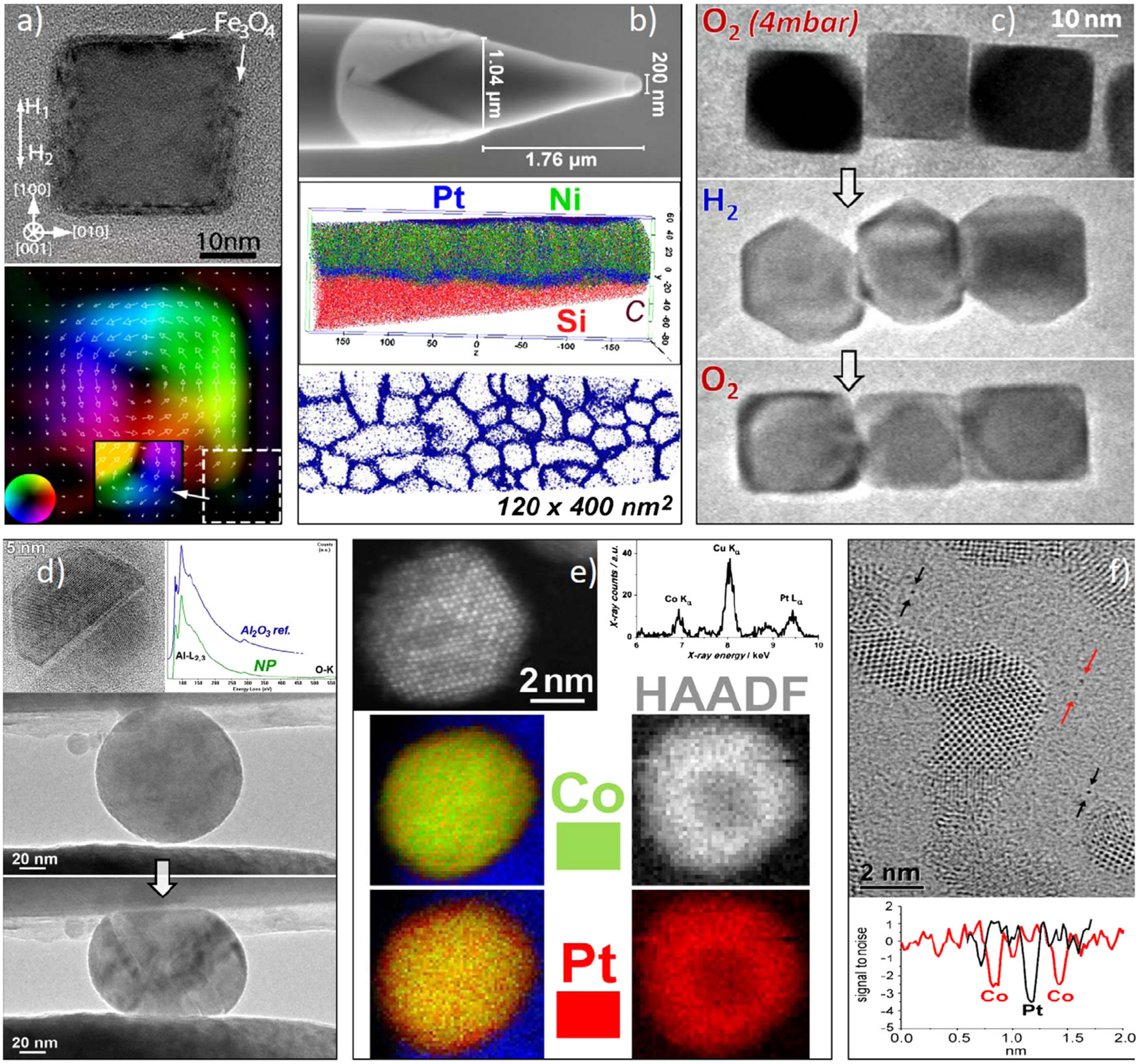

Fig. 1. (Colour online.) Illustration of the expertise of the METSA members: a): holography and Lorentz EM study of the magnetic configuration in 30-nm Fe nanocubes (CEMES) [5]; b) APT study of the segregation of Pt at the $NiSi_2$ grain-boundaries in a Ni silicide alloyed with Pt (IM2NP) [6]; c): evolution of morphology of Pt nanoparticles under gas environment followed by in situ TEM (CINAM) [7]; d): EELS and in situ TEM compressive measurements of transition alumina nanoparticles (CLYM) [8]; e): STEM-EELS imaging of CoPt catalysts; Co and Pt maps shown on the left are respectively for the same fresh (top) and aged (bottom) nanoparticle (PFNC) [9]; f): CoPt nanoparticles deposited on a carbon film: detection of individual Co and Pt atoms by C_s-corrected HRTEM (MPQ) [10]. g): atomic STEM-EELS study of a $BaTiO_3$–Fe tunnel junction (LPS) [11]; h): 3D analysis of the crystallography of K–Ta–Nb–O tetragonal tungsten bronze nanorods by precession electron diffraction (CRISMAT) [12]; i): APT and 3D field ion microscopy analysis of atomic NbN platelets in ferrite (GPM) [13]; j): TEM nano-tomography of a alumino-silicate zeolite (IPCMS) [14]. (*Continued on next page.*)

According to these goals, METSA has provided the access to more than 500 researchers for 392 projects over more than 480 deposited, representing 1400 days during the five years of existence of the network. These experiments were initiated in a great majority by French teams, but a dozen of projects were also proposed by international groups as well. The origin of the demands for METSA experiments is illustrated in Fig. 2; interestingly, it shows that physics, chemistry and engineering applications are the three main and roughly equivalent areas for which the access to advanced METSA instruments is requested.

More facts and features

The eight METSA platforms offer an access to three atom probes and 17 electron microscopes, mainly aberration-corrected TEMs, but also one dedicated environmental SEM and 3 double-column SEM–FIB. Some of these instruments are among the most advanced generations, i.e. the aberration corrected Nion Ultra-STEM 200 kV in LPS Orsay, the cold-FEG JEOL ARM in MPQ Paris, the double-C_s corrected and monochromated FEI TITAN 'Ultimate' in PFNC-CEA Grenoble, the cold-FEG HITACHI HF3300C 300 kV in CEMES Toulouse, the laser Flextap CAMECA Atom Probe in Rouen, and soon the C_s-corrected Environmental TITAN ETEM 300 kV in CLYM Lyon-Villeurbanne.

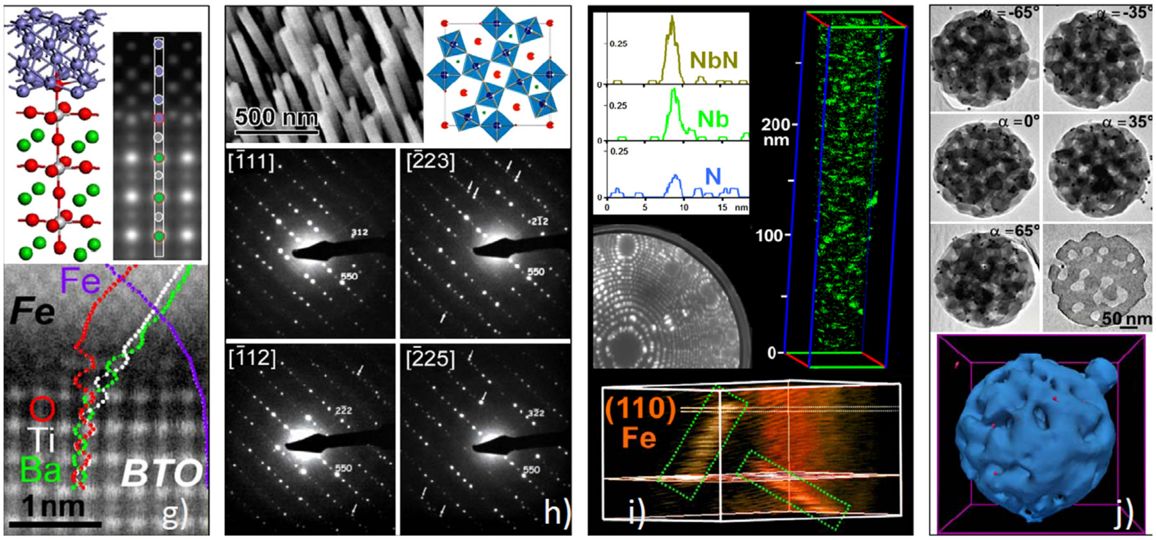

Fig. 1. (*continued*)

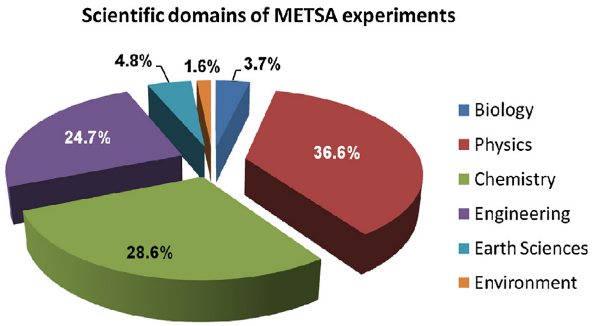

Fig. 2. (Colour online.) Scientific repartition of METSA experiments since 2009.

All these instruments have contributed to the experiments conducted so far, which have led to more than 50 published papers and about twice communications to international meetings and conferences, as well as significant contributions to more than 10 theses. In addition to the direct support to research through taking care of scientific experiments, METSA aims at participating to training actions at the French and European levels; as first initiatives, METSA has supported the second edition of the QEM (Quantitative Electron Microscopy) international school[2] in 2009, and more recently the training courses on electron tomography organized in Strasbourg in 2012[3] in relation with the French Society for Microscopies (SFμ).[4]

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