Atom Probe Tomography experiments performed in a (Scanning) Transmission Electron Microscope

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Background

Atom Probe Tomography (APT) is intrinsically a 3D characterization technique that provides tomographic reconstructions of materials with a near atomic scale resolution. For APT, specimens must be prepared as sharp needles. This is most of the time realized using FIB SEM and before analysis in APT, specimens can easily be characterized in a TEM or STEM using specific holders or protocols. Hence, correlative analysis by (S)TEM and APT gives access to a wide range of information about the specimen. Unrivaled spatial resolution of (S)TEM, availability of 4D-STEM or diffraction with the combination of the 3D composition fields accessible by APT hence leads to a better description of materials by the correlation of structural, physical and chemical characterization.

To make correlative analysis by (S)TEM and APT more accessible, efforts have been foreseen and made to join both techniques in a single instrument [1-4]. This work presents the main achievement of an instrumentation project started at GPM in 2014, which is the implementation of an Atom Probe in a commercial JEOL F2 (Scanning) Transmission Electron Microscope.

Methods

Specifics TEM holders were designed first for a JEOL 2010 TEM, on which they were tested, and then transferred to a JEOL F2 installed at University of Rouen in 2022. These holders offer the possibility to perform APT experiments either at room temperature or cryo temperature (78K measured at the tip). They accept APT specimens which can be polarized with a positive voltage up to 8 kV. Electrostatic pulses are superimposed to trigger field evaporation with a pulse repetition rate of 20 kHz. The APT detector consists in an advanced-delay line setup, mounted on a port facing the goniometer of the microscope.

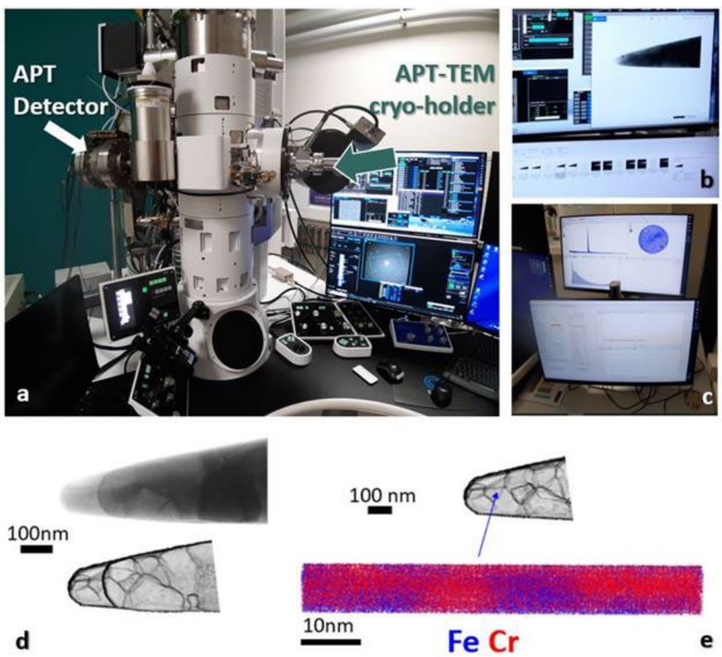
Results

The figure below shows the setup realized in the framework of this project. The ion sensitive and time resolved spatial detector is mounted behind the column (on the left in the image) and APT specimens are loaded classically in the goniometer, using home designed holders. All imaging conditions available with the microscope can be applied to characterize APT specimens. Here, an illustration is given with the automated crystal orientation mapping method. A nearly equiatomic Fe-Cr alloy with an ultrafine grain structure has been submitted to a heat treatment leading to spinodal decomposition. The visualization of grain boundaries in the APT specimen allows to localize unambiguously the location of APT analysis in the specimen.

Conclusion

This new instrument, combining APT with (S)TEM, opens up new prospects for unambiguously correlating the characterization of structural defects (such as grain boundaries, precipitates or dislocations) identified in TEM with the chemical characterization, atom by atom, accessible in tomographic atom probes.

Graphic:



(a) JEOL F2 microscope equipped with an APT-TEM cryo-holder and APT detector both designed at GPM. (b) BF-STEM of a specimen and (c) sequence of acquisition of an Al-alloy. (d) Superimposition of images acquired before and after APT acquisition in JEOL F2. Top images are BF-STEM and images below show grain boundaries imaged with the ASTAR software of an ultrafine grain FeCr alloy. (e) APT reconstruction of the same alloy, showing the region of interest analyzed in APT.

Keywords:

Atom Probe Tomography, STEM, instrumentation

Reference:

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