Nuclearization projects of a tomographic atom probe and of an electropolishing machine for researches on neutron irradiated materials at the atomic scale

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The Tomographic Atomic Probe (TAP), located in LECI, in the CEA/Saclay LECI hotlab facilities, has become an essential technique for the fine characterization of nuclear materials. Indeed, this high-resolution three-dimensional microscope is a unique instrument because it allows an understanding of the phenomena at very fine scales namely the atomic scale with unparalleled chemical and spatial sensitivity, hence it presents a major interest for the characterization of defects created by irradiation that are mostly visible at these scales. In the aim of studying neutron-irradiated materials with this instrument, LECI undertook the nuclearization project of the TAP. This project, which is a major stake for LECI, includes several milestones, namely the manufacturing of a glove box, the instrumental nuclearization, and then the nuclearization of the rooms (engineering studies and building works to accommodate irradiated samples) associated with the nuclear safety and radiation protection studies of this project. As TAP needs specific samples (with a needle shape) to be studied, this project is associated to the nuclearization project of a sample preparation machine.

Nuclearization project of a tomographic atom probe

This project is part of the “Equipement d’Excellence” called GENESIS[2],[3] that is a characterisation platform dedicated to the analysis of irradiated materials at nanometric scale where LECI is associated to GPM (Rouen, France) and CIMAP (Caen, France). Within this project, LECI is currently installing a TAP (and a FIB/SEM which will not be presented here). Scheduled to be operational at the end of 2018, this instrument will allow observing irradiated nanometric samples in order to improve safety studies associated to the aging of actual and new material used in nuclear power plants, and to study the degradation mechanisms produced by neutron irradiation at atomic scale.

To complete this project, several actions were carried out. The first one consisted in the instrumental nuclearization that is it to say the modification of the TAP itself and the manufacturing of a glove box.

The modification of the TAP including the connection of the TAP to a device for vacuum transfer of samples and its docking port, the shielding of the detector to minimize background noise, the remote handling of the vertical rod and a system to recover fallen irradiated samples in the analysis chamber. – CAMECA supplied these changes (see Figure P27).

[2] This work has benefited from a state aid managed by the National Research Agency under the program "Investments for the future" with the reference ANR-11-EQPX-0020

[3] Groupe d’Etudes et de Nanoanalyses des Effets d’Irradiations

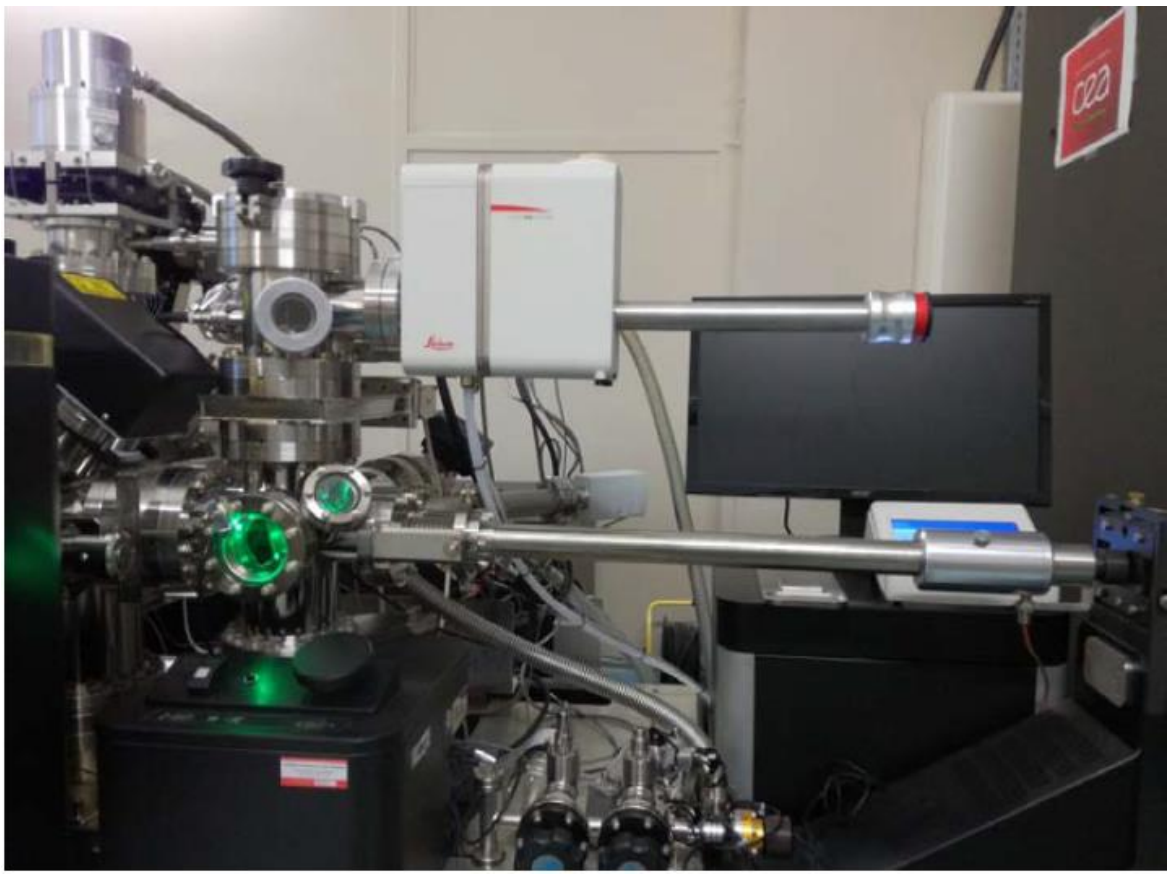

Figure P27: The nuclearized TAP

A glove box was also supplied (by DEFI SYSTEMS) ensuring many functions as (see Figure P28):

- the reception of the neutron irradiated samples from the sample preparation machines (electopolishing or MEB/FIB),
- the storage of the irradiated samples in a vacuum shielded cabinet,
- the preparation of the samples on the holders appropriated to the TAP (or the TEM),
- the possibility to clean samples and holders with a plasma cleaner.

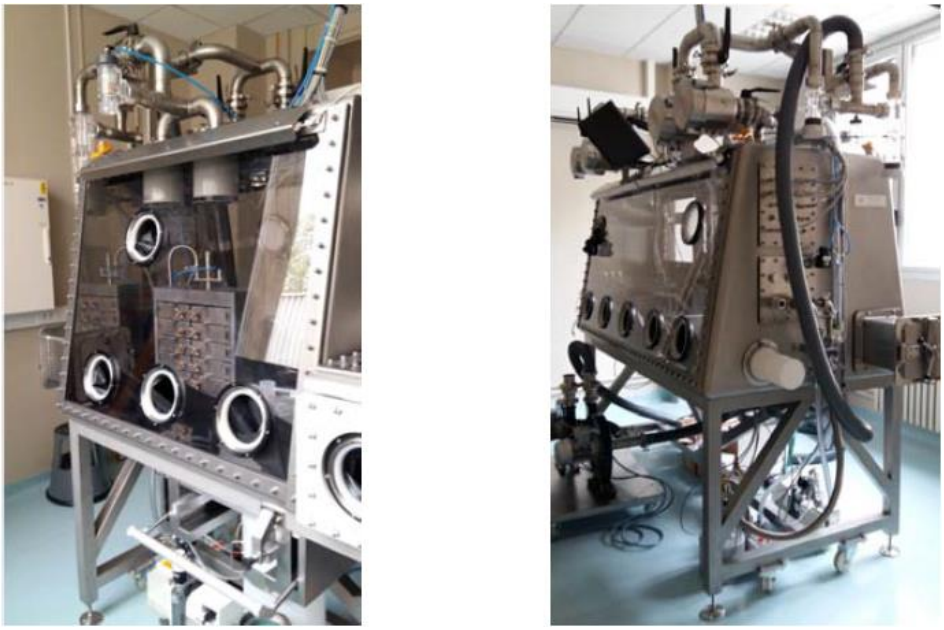

Figure P29: The TAP glove box

Another important point of the project was to nuclearize the rooms that will host the nuclearized TAP and the glove box. Many engineering studies and building works were conducted with the collaboration LECI operating team. Moreover, nuclear safety and radiation protection studies were realized to obtain the authorization from the regulator to analyse neutron-irradiated materials with the TAP. The final decision of the regulator is expected by the end of 2018.

Nuclearization project of a sample preparation machine

A TAP study requires a special preparation of the samples, because they must be fabricated in the form of a needle with an end radius between 10 and 100 nm. Electropolishing is one of the methods used for this particular sample preparation. Its principle is to chemically smooth a sample of 1 cm long and approximately 0.25 mm^2 or circular in cross section to obtain a needle (**Figure P30**).

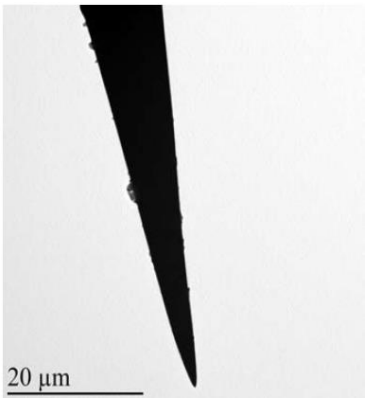

Figure P30: A needle for a TAP study

To characterize irradiated samples with the TAP, the laboratory of microscopy of the LECI hot lab studied and designed an electropolishing machine to be integrated in a glove box dedicated to the preparation of samples with chemical products (see Figure P31).

Figure P31**:** The glove box for the electopolishing machine

The electropolishing machine is composed of many elements that is to say the mechanical part and electronic modules (power generator and motion controller). Moreover, the laboratory developed a software for the control of the machine with LabVIEW. A picture of the software interface is visible in Figure P32.

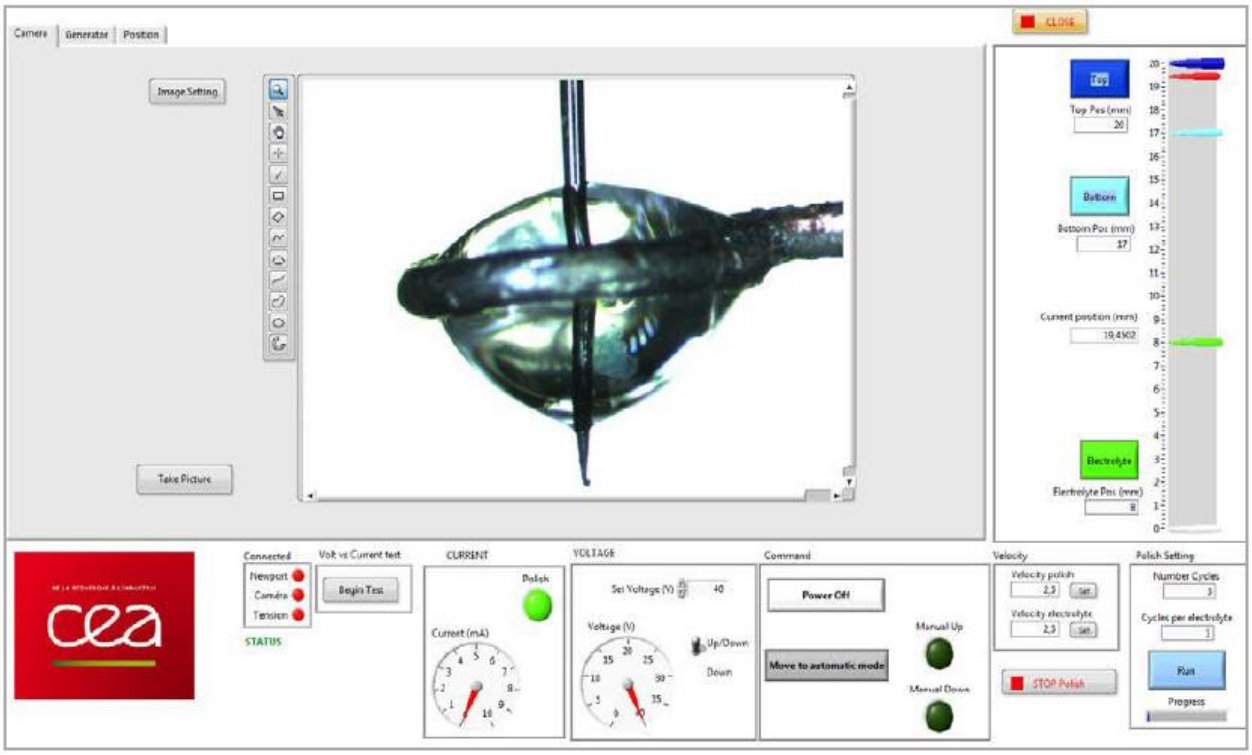

Figure P32**:** Print screen of the interface of the electopolishing machine software developed by CEA

The next step for this project is to integrate the electropolishing machine in the dedicated glove box with suitable connectors. The hardware and relevant electronic modules will be implemented in the room out of the glove box. It will be done by September 2018.