

Report about the first operative half-year of the JEOL JEM-F200 Multipurpose HR-TEM

General overview and management

The TEM was delivered on July 31st 2021, but already in September appeared a critical failure with the Gatan CMOS Camera. The problem almost completely hindered the use of the TEM up to December 2021. As a consequence, JEOL agreed to postpone the effective beginning of the guarantee to January 1st 2022, a guarantee that extends up to December 31st 2022 and will be followed by one year of free maintenance contract (partial assistance).

The TEM was under the responsibility of Prof. Simone Capaccioli, Director of CISUP, and Prof. Luca Pandolfi, Director of Earth Science Department of UniPI, till March 2022. In March 2022, Prof. Enrico Mugnaioli was appointed RAR and RAD of the TEM lab and designated as responsible of the TEM by CISUP. The latter position was ratified by CISUP Council in August 2022.

In April a Management Committee was also designated by CISUP, including Prof. Vincenzo De Tata, Prof. Leonardo Rossi and Prof. Renzo Valentini. The Management Committee was also ratified in August 2022, with the additional inclusion of Prof. Vittoria Raffa.

The activities at the TEM lab are monitored by this Committee since March 2022.

TEM operational activities

TEM lab was continuously working from March to June 2022. At the beginning of July 2022 a second failure with the Gatan CMSO camera occurred. For most of July, the TEM could be used only for those activities that did not require the CMOS camera.

In August 2022 the TEM was switched in eco-mode and not operated.

At the beginning of September 2022 a failure with the goniometer stage occurred, resulting in a stop of two weeks. From September 21st till September 23rd, the TEM had its year revision, with no further issue reported by the JEOL technical staff.

Users

The current habilitated independent users are Prof. Enrico Mugnaioli (Department of Earth Sciences, University of Pisa) and Dr. P. Lucchesi (Department of Clinic and Experimental Medicine, University of Pisa). Both of them are also superuser and in charge for the training and the assistance of future users.

Use of the machine

The TEM lab has been operated for 39 days, roughly equivalent to 312 hours. In this first period free-of-charge measurements were encouraged, with the aim to promote the establishment of future collaborations with the Departments of UniPI and with other Italian and foreigner research institutions, as detailed below.

Three collaborations produced or are supposed to produce an income for the TEM lab.

TEM was also used for training and didactic activities.

The use of TEM is detailed in the following table and graphs.

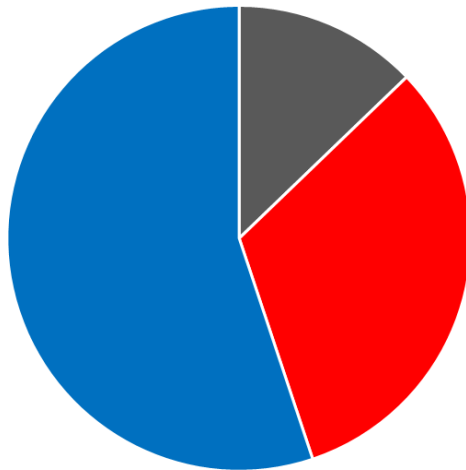
Days	Type of activity	Operator	Description
3	Lab Management	Mugnaioli E.	Calibrations and check of TEM performances
2	Lab Management	Lucchesi P.	Check of TEM performances
3	Training	Lucchesi P.	Training of Dr. Modeo (DB) and Dr. Scavuzzo (UniPi- DRTNTMC)
2	Training	Mugnaioli E.	Training of Dr. Lucchesi (UniPI-DMCS)
1	Training	Mugnaioli E.	Training of Dr. Parlanti (IIT-CMI@SSSA)
1.5	Didactic activity	Mugnaioli E.	Electron Microscopy of Nanomaterials – Dr. Gemmi, Materials and Nanotechnology
3.5	Didactic activity	Mugnaioli E.	Principi ed applicazioni pratiche del microscopio a trasmissione elettronica (TEM) – Dottorato Regionale in Scienze della Terra
1.5	Thesis and internship	Mugnaioli E.	Analysis of nanowires for electronic applications – Thesis of Mr. Neri (UniPIDF and CNR-IN)
4	Financed project / collaboration	Mugnaioli E.	Analysis of keu particulate – Collaboration with Prof. Petrini (UniPI-DST) and ARPAT
0.5	Financed project / collaboration	Lucchesi P.	Analysis of wild type and mutant <i>Bacillus cereus</i> through negative staining – Collaboration with Prof. Ghelardi (UniPI-DRTNTMC)
2	Financed project / collaboration	Mugnaioli E.	Structural characterization of nanocrystalline CaCO ₃ seeds – Collaboration with Prof. Nemeth (Hungarian Center for Natural Sciences)
1	Free-of charge test measurement / collaboration	Mugnaioli E.	Characterization of black sand – Collaboration with Prof. V. Castelvetro (UniPI-DCCI)
1	Free-of charge test measurement / collaboration	Mugnaioli E.	Characterization of impact droplets – Collaboration with Prof. Folco (UniPI-DST) and Dr. Gemmi (IIT-CMI@SSSA)
3	Free-of charge test measurement / collaboration	Mugnaioli E.	Structural characterization of modulated bronzes and planerite mineral – Collaboration with Prof. Bonazzi (UniFI)
1	Free-of charge test measurement / collaboration	Mugnaioli E.	Analysis of asbestos – Collaboration with Prof. Gualtieri (UniMORE)
1.5	Free-of charge test measurement / collaboration	Mugnaioli E.	Characterization of nanoparticles and microfibers – Collaboration with Dr. D'Acunto (CNR-IB)
1	Free-of charge test measurement / collaboration	Mugnaioli E.	Analysis of core-shell nanoparticles – Collaboration with Dr. Voliani (IIT-CNI@NEST)
1.5	Free-of charge test measurement / collaboration	Mugnaioli E.	Analysis of hydrothermal cronstedtite samples – Collaboration with Prof. Pignatelli (University of Nancy)
3	In-house measurements	Mugnaioli E.	Ultra-reduced micro-inclusions in Tibet ophiolites
2	In-house measurements	Lucchesi P.	Algae symbionti and flagellates

Type of activity



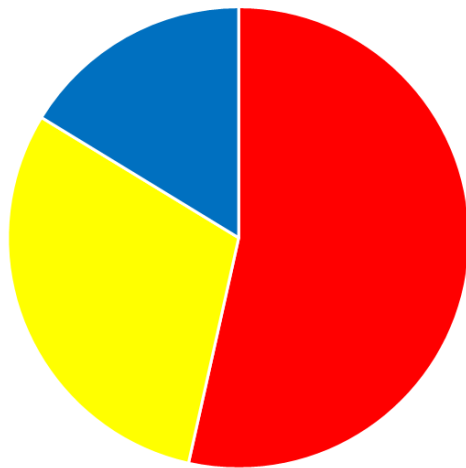
- Lab Management
- Training
- Didactic activity
- Thesis and internship
- Financed project / collaboration
- Free-of charge test measurement / collaboration
- In-house measurements

Type of activity



- Lab Management
- Formation
- Research

Type of scientific collaboration



- UniPi
- Other Italian research institutions
- Foreigner research institutions

Products

Data collected by the TEM are reported in a number of papers submitted to international peer reviewed papers or currently under preparation.

Data collected are also reported in the following congress communications:

- 1) Mugnaioli E.: **3D electron diffraction applied to complex nanoparticles and nanominerals.** Annual Meeting Swiss Society for Crystallography, Bern (Switzerland), September 2022 (*invited lecture*).
- 2) Mugnaioli E., Folco L., Masotta M., Biagioni C., Paoli G., Capaccioli S., **The new CISUP facilities for Earth Science at the University of Pisa.** Geosciences for a Sustainable Future, Turin (Italy), September 2022.
- 3) Perchiazzi N., Mugnaioli E., Pollastri S., Franceschini F., Licitra G., Ghezzi L., Petrini R.: **Solid-state characterization of high-temperature processed chromium-bearing tannery sludges.** Geosciences for a Sustainable Future, Turin (Italy), September 2022.
- 4) Mugnaioli E., Xiong F., Xu X., Yang J., Grew E.S.: **η -phase Ti_2Fe and κ -phase $Ti_{10}Fe_3$: 3D ED study of potential new minerals from Tibet, China.** 23th General Meeting of the International Mineralogical Association, Lyon (France), July 2022.
- 5) Mugnaioli E.: **Structure characterization of $CaCO_3$ in early formation stages by 3D electron diffraction.** Geochemistry and Mineralogy of Calcium Carbonate Polymorphs, Veszprém (Hungary), July 2022.

Finally, the data were also used for the proposal of two new minerals to the Commission on New Minerals, Nomenclature and Classification (CNMNC) of the International Mineralogical Association (IMA).

Income and outcome of the TEM lab

The collaboration with Prof. Petrini (UniPI-DST) and ARPAT for the analysis of keu particulate was worth for an income of 10.000,00 €

The collaboration with Prof. Nemeth (Hungarian Center for Natural Sciences) for the analysis of nanocrystalline $CaCO_3$ seeds was worth for an income of 4.000,00 €

The collaboration with Prof. Ghelardi (UniPI- DRTNTMC) for the analysis of stained viruses is supposed to bring an outcome of about 1.000,00 €

The mission of Prof. Mugnaioli to the Geochemistry and Mineralogy of Calcium Carbonate Polymorphs costed 540,00 €

Consumables and cryogenic gas for the TEM lab costed 6.586,40 €

The current available leftover is 10.783,00 €

Forecast technical improvements

We forecast the connection with the UniPi Data center in the next few months. We also forecast the installation of a new screen fully dedicated to the ASI computer.

CISUP council approved the acquisition of an ion miller that will facilitate sample preparation from bulk inorganic materials. This upgrade will facilitate the access to TEM for scientist working on bulk inorganic materials (alloys, rocks, precipitates) and on surfaces.

We also expect a beneficial interaction with the Dual-Beam facility recently installed at the Department of Physics by CISUP.

We also aim for the acquisition of a multi-sample holder for speeding routine analyses of both biologic and inorganic samples.

Open issues and future perspectives

The TEM lab is already producing interesting results, but is still not using the full potential of the Multipurpose F200 TEM. In particular, HR-STEM, SerialEM and ASTAR capabilities have not been used.

The main issue is the lack of users. In this regard, we ask CISUP to promote inside UniPI a campaign for raising awareness about the capabilities of the TEM lab for material and tissue characterization.

Increasing the number of users may comprise:

- Dedicated CISUP technical units trained for TEM, which can perform routine measurements, lab maintenance, user supervision, sample preparation and lab administration;
- The identification of dedicated technical or research units in each Department of UniPI which may be dedicate part-time to TEM activities, with the aim to become a reference person in their Department for TEM measurements and investigations.
- Already-formed or to-be-formed scientific staff working in the area of Pisa and belonging to research institution outside UniPI (CNR, IIT, SNS, SSSA, AOUS).
- PhD students from different Departments whose doctoral project include TEM activities.

Current TEM users will be happy to train all this staff. We evaluate in 10 to 15 the optimal number of TEM habilitated users. Also, the collaboration with CIME must be enforced and better articulated.

Beside TEM users, we also stress the need of a dedicated technical unit for the preparation of biologic samples. The collaboration with Dr. Modeo has been fruitful, but will soon end because Dr. Modeo is to become RTD-B at UniPI.

We also ask CISUP to encourage the establishment long-lasting collaborations between the TEM lab and the Departments of UniPi or other research institutions, with a fix cost per year and a fix dedicated TEM time per week. Such collaborations will give a solid base for the self-maintenance of the TEM lab.

In this regard, we are working for the establishment of collaborations with Life Science Departments, DICI, AOUS, CNR-IB and CNR-IN. We hope that other collaborations will be established in the next future.

We are also working for establishing a collaboration with the Belgian company NANOMEGAS, which asked to use our TEM lab as a demo-lab for ASTAR software. Beside a reliable financial income, this collaboration would also assure the TEM lab to collaborate with the best experts in phase/orientation diffraction mapping.

Finally we point out the value of the on-going free-of-charge collaboration with Dr. Gemmi (IIT-CMI@SSSA), which allows the TEM lab to collaborate with one of the most internationally recognized research group on electron crystallography.