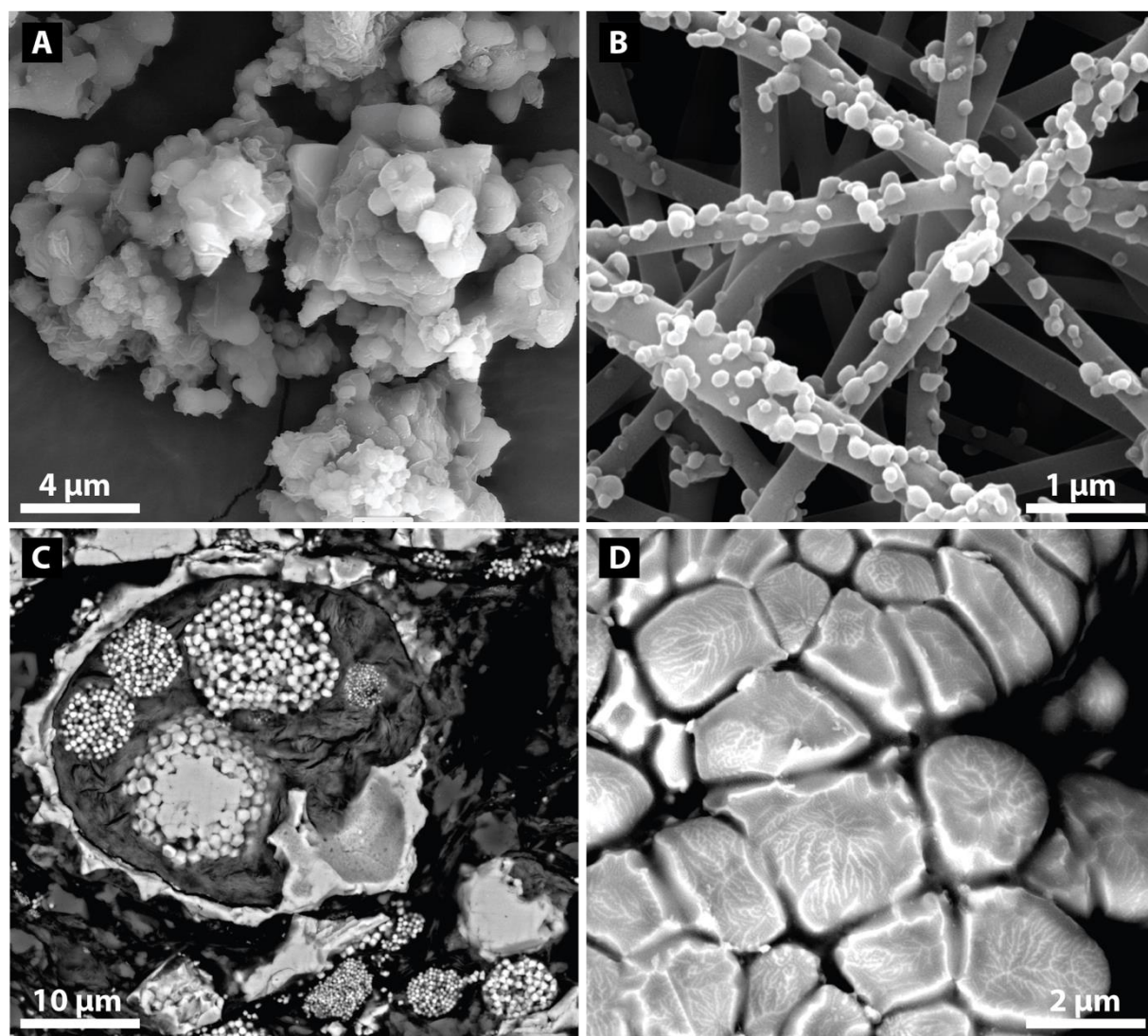


# CISUP ESEM-FEG laboratory: 2020 activity report

Luigi Folco, Randa Ishak, Gabriele Paoli, Maurizio Gemelli, Massimo D'Orazio and Andrea Lazzeri

Laboratory web-page: <https://cisup.unipi.it/labs/FEI-Quanta-450-ESEM-FEG>



Cover image: A) Synthesized  $\text{Li}_4\text{SiO}_4$  powders for  $\text{CO}_2$  capture at high temperature. Secondary electron image (SEI). Courtesy of Maurizia Seggiani. B) Poly(ethylene oxide terephthalate)-poly(butylene terephthalate) (PEOT/PBT) electrospun fibers functionalized with electrosprayed Rhodamine-loaded-PLGA nanoparticles (SEI). Courtesy of Serena Danti and Bahareh Azimi. C) Framboidal pyrite and pyritized micro-fossils embedded in a quartz + chlorite matrix. Cretaceous black shale from the "Scaglia toscana" formation, Deccio di Brancoli, Lucca Province. Back scattered electron image. Courtesy of Massimo D'Orazio. D) Nanodendrites of Fe,Ni-oxides on the external surface of an impact melt spherule from the Kamil crater, Egypt. Courtesy of Luigi Folco.

## CISUP ESEM-FEG laboratory: 2020 activity report

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*Abstract* – Through this annual report we would like to inform the UniPi community on the activity carried out by the CISUP ESEM-FEG laboratory during the year 2020. We report here on: i) laboratory implementation; ii) new price list and educational package; iii) hours worked; iv) user statistics; v) research products; vi) outreach; vii) issues and objectives. The installation of the LEICA EM ACE600 high-vacuum sputter coater, of the Ar and N<sub>2</sub> gas lines and the remote access system are the main lab developments achieved in this year. Despite the ongoing pandemic, the lab managed to work for a good 290 hours, equal to about 2/5 of the average annual budget. The lab has nevertheless provided service to tens of researchers and PhD/MSc students from 5 departments of our University, and few third parties, including the *Legione Toscana dei Carabinieri*. The work carried out in the lab is acknowledged in 36 papers published in international journals with IF. Contrary to the previous years, the laboratory required financial support from CISUP to cover the costs of the annual maintenance contract. This was in part due to the pandemic, in part to the pending lab credits (with a number of academics who co-funded the purchase of the instrument in 2016) the current management inherited. Increasing the performance of the lab to bring the numbers of hours worked back to the sustainable figure of ~700 – 800 hours/year and extending the usership to other departments (particularly life sciences and humanities) is our top priority for year 2021. This report will also be publicly available through the CISUP web page (<https://cisup.unipi.it/>).

### 1. Laboratory implementation

**1.1. Coatings - High-vacuum sputter coater.** On January 23<sup>rd</sup>, the Leica EM ACE600 sputter-coater has been successfully installed in our lab by Leica staff. The Leica EM ACE600 is a versatile, state-of-the-art, high-vacuum film deposition instrument for precise coating of samples for both FE-SEM and TEM applications. Two angled sources are configured. One for metal sputtering (Au and Pt targets are currently available) and one for C-thread evaporation. An automated stage rotation is integrated for best distribution of the coating. An integrated quartz crystal measurement accurately determines the layer thickness. Tilt and height can be set manually. Any material can be processed as long as it is not sensitive to vacuum, argon plasma or the heat generated during carbon coating. The vacuum system creates an ultimate vacuum  $\leq 2 \times 10^{-6}$  mbar enabling even distribution of the coat even in porous samples.

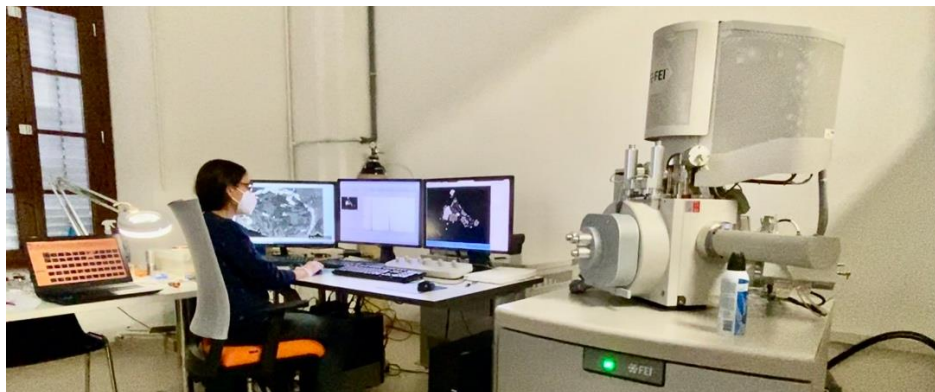
There is no charge for coating samples to be analyzed in our laboratory.

The instrument was acquired by CISUP, thanks to a donation-based crowdfunding of a bunch of CISUP volunteers from the Dipartimento di Scienze della Terra (DST), the Dipartimento di Fisica (DF), the Dipartimento di Chimica e Chimica Industriale (DCCI), the Dipartimento di Ingegneria Civile e Industriale (DICI) and our laboratory itself.



**Figure. 1.** The Leica EM ACE600 sputter-coater at the CISUP ESEM-FEG lab.

**1.2. Remote access system.** In November 2020, we developed a simple IT system to share the two screens of the electron microscope via internet. The system allows a remote user to instruct an operator in the laboratory in running his/her own analyses. The system makes use of the TEAMS platform. For the scope, a third computer was purchased. The third computer acts as a “firewall” to protect the software controlling the microscope. Beside the potential of widening the usership in normal times, the system proved very effective during the ongoing pandemic, in compliance with the safety restrictions.



**Figure. 2.** The new display set up at the CISUP ESEM-FEG lab.

The IT system was developed thanks to technical support of M. Savini, S. Avino and D. Armaioli of the Sistema Informatico di Ateneo. Proudly we report that the system was first tested by prof. Paolo Mancarella, Chancellor of the University of Pisa, and prof. Simone Capaccioli, director of CISUP, with the *in situ* technical assistance of one of us (RI).

Funding for the IT system was provided by CISUP (*fondi funzionamento*).

**1.3. Ar and N<sub>2</sub> gas lines.** In late December 2020, the Ar and N<sub>2</sub> gas lines have been installed in our lab by the SOL Group company, in compliance with UniPi safety regulations. The two lines serve the Leica EM ACE600 sputter coater and the FEI QUANTA 450 FEG sample chambers, respectively. They are supplied by gas cylinders placed in newly installed outdoor storage box located outside the Palazzina Piaggio.

Funding was provided by CISUP (*fondi sicurezza/funzionamento*).

## 2. New price list

Table 1 shows the update price list of the ESEM-FEG FEI QUANTA 450 laboratory, for routine analyses. These include secondary electron (SE) and back-scattered electron (BSE) imaging, EDX microanalyses/mapping, scanning transmission electron microscopy (STEM) analyses and electron back-scattered diffraction analyses (EBSD).

**Table 1.** CISUP ESEM-FEG laboratory: Prices for routine analyses including SE and BSE imaging, EDX, STEM and EBSD. For overnight mapping, in situ HT and LT experiments please contact laboratory staff for a quotation.

<i>Academics</i>		
Teaching package*	20 €/hr	
Experts	30 €/hr	
Non-experts with technical assistance	50 €/hr	
Non-institutional work	70 €/hr	
<i>External</i>		
CISUP fellow	50 €/hr	(+IVA)
Research Institutes	70 €/hr	(+IVA)
Privates	100 €/hr	(+IVA)

\*Includes course practicals and up to 3 ESEM-FEG sessions (up to 3 hours each) for MSc theses assisted by his\her tutor (if qualified as expert).

Basic fares have not changed since the start for the lab in fall 2016, but we have now two additional options. The first one is the educational package, which includes course practicals and up to 3 ESEM-FEG sessions (up to 3 hours each) for MSc theses assisted by his\her tutor (if qualified as expert). The second is for non-institutional work carried out by UniPi academic staff for third parties.

## 3. Hours worked

Table 2 shows a breakdown of the hours worked by the ESEM-FEG laboratory since its establishment in fall 2016. During year 2020, the laboratory worked for 290 hours. This is about 2/5 of the average amount of hours (~700) worked during the previous three years. As a result, the gross 2020 income is ~14.6 k€ against the 2017-19 average value of ~34.3 k€.

**Table 2.** ESEM-FEG usage (2020.12.31 update) in terms of hours (blue) and equivalent income (black).

Time period	Sep-Dec 2016		2017		2018		2019		2020		Totals	
	hrs	€eq	hrs	€eq	hrs	€eq	hrs	€eq	hrs	€eq	hrs	€eq
Non experts (50 €/hr)	65.5	3275	436	21800	555	27750	467	23325	258	12900	1781	89050
Experts (30 €/hr)	45.5	1365	188	5640	235	7043	239	7170	8	240	715	21448
Non-institutional work (70 €/hr)									1	70	1	70
Other research institutes <sup>a</sup>							2	140			2	140
External/private (100 €/hr)			35	3500	27	2700	38	3800	14	1400	117	11700
<i>Pro bono</i>									9	0	9	0
<b>Totals</b>	<b>111</b>	<b>4640</b>	<b>659</b>	<b>30940</b>	<b>820</b>	<b>37473</b>	<b>746</b>	<b>34435</b>	<b>290</b>	<b>14610</b>	<b>2626</b>	<b>122408</b>

Note that, in late July, the Bruker EDX detector broke. The detector module was replaced by Bruker and Thermo technical staff at the end of October. As a result, unfortunately, several user applications for microanalytical work could not be satisfied in the meanwhile. The costs of the EDX detector (~10 k€ + IVA) were covered in full by the THERMO Full-risk annual maintenance contract.

### 3. User statistics

The users of the ESEM-FEG laboratory over the year 2020 include 38 research groups from 5 departments of the University of Pisa (DICI, DCCI, DST, DF, and DFARMA) accounting for ~92% of the total workload; the remaining ~8% consisted of services for privates and *pro bono* activities (Fig. 3). The department with the largest number of involved research groups is DICI (18) followed by DCCI (8), DST (6), DF (2) and DFARMA (1). The DICI boasts the largest volume of laboratory work (~45% of the total), followed by DCCI (20%), DST (15%), DF (~3%) and DFARMA (1%). Overall, the statistics is similar to that of the previous ~2.5 years. *Pro bono* activities are a welcome addition to our current stat. The laboratory actually worked for a forensic study for the *Legione Carabinieri Toscana*. Noteworthy, also, the investigation of the *Ponte Morandi* in Genoa and a quality control on *PPE and face masks* for the coronavirus pandemic benefited of analyses carried out in our laboratory. On the downside, and contrary to the previous years, we could not carry out educational practicals and outreach laboratory work due to COVID restrictions, which comprised 5% and 1 % of the total workload, respectively.

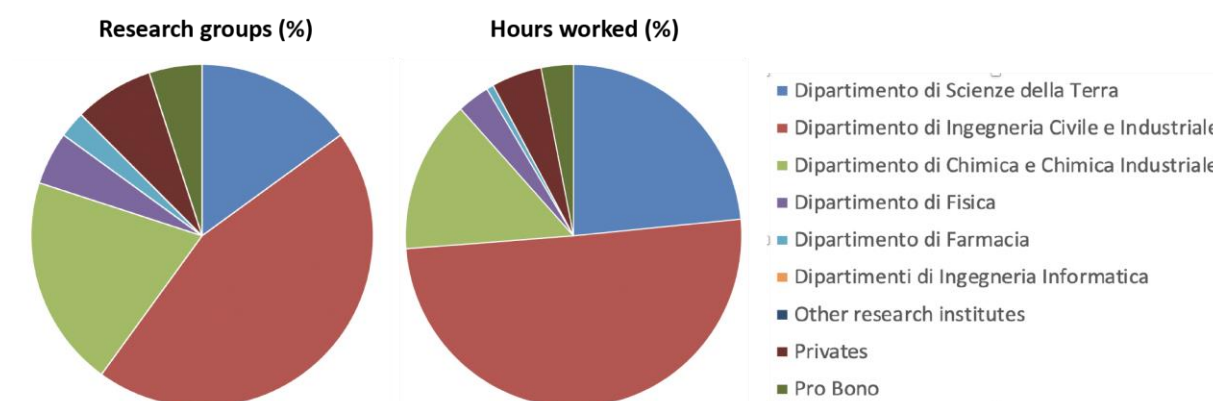


Fig. 3. Laboratory user statistics – year 2020.

### 4. Research products

In December 2020 we asked the ESEM-FEG laboratory users to provide us with a synthetic list of academic and technological products (publications, book chapters, patents, thesis, etc.) reporting works which benefited of our laboratory and published in 2020. Several research groups from DCCI, DCI, DST and DF responded promptly.

The outcome of the above research groups is remarkable with **36 peer-reviewed papers**, including one published by *Nature Publishing Group*. Below is the list of the 2020 products received. We hope to implement the list of products below in the future with the collaboration of all lab-users.

#### Research article list (peer-reviewed only)

1. Aliotta L, Gigante V, Acucella O et al. (2020) Thermal, mechanical and micromechanical analysis of PLA/PBAT/POE-g-GMA extruded ternary blends. *Frontiers in Materials*. <https://doi.org/10.3389/fmats.2020.00130>
2. Aliotta L., Gigante V., Cinelli P., Coltelli M.B., Lazzeri A. (2020) Effect of a Bio-Based Dispersing Aid (Einar® 101) on PLA-Arbocel® Biocomposites: Evaluation of the Interfacial Shear Stress on the Final Mechanical Properties. *Biomolecules*, 10, 1549. <https://www.mdpi.com/2218-273X/10/11/1549>
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7. Biagioni C., D'Orazio M., Fulignati P., George L. L., Mauro D., Zaccarini F. (2020) Sulfide melts in ore deposits from low-grade metamorphic settings: Insights from fluid and Ti-rich sulfosalt microinclusions from the Monte Arsiccio mine (Apuan Alps, Tuscany, Italy). *Ore Geology Reviews* 123, 103589. <https://doi.org/10.1016/j.oregeorev.2020.103589>
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10. Coltelli M.B., Aliotta L., Vannozzi A., Morganti P., Panariello L., Danti S., Neri S., Fernandez-Avila C., Fusco A., Donnarumma G., Lazzeri A. (2020) Properties and Skin Compatibility of Films Based on Poly(Lactic Acid) (PLA) Bionanocomposites Incorporating Chitin Nanofibrils (CN). *Journal of Functional Biomaterials*, 11, 21. <https://doi.org/10.3390/jfb11020021>
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19. Dionnet Z, Suttle M. D., Longobardo A., Rotundi A., Folco L., Della Corte V., King A. (2020) X-ray Computed Tomography: morphological and porosity characterization of giant Antarctic micrometeorites. *Meteoritics & Planetary Science* 55,1581 – 1599. <https://doi.org/10.1111/maps.13533>

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## 5. Outreach

On 2020, November 27 the ESEM-FEG FEI Quanta 450 laboratory was presented to the public by prof. Simone Capaccioli, director of CISUP. This occurred during a virtual tour of the Research Centers of the University of Pisa, organized for the “2020 *BRIGHT-NIGHT la Notte europea delle ricercatrici e dei ricercatori*” event.

## 6. Issues and objectives

**5.1 Performance.** Most of the work carried out so far has been focused on SEM imaging and EDX micronalysis (both in environmental and high/low vacuum modes), with limited usage of the STEM system, and practically no use of the EBSD system and the heating and Peltier stages. Too bad! We seek researchers with expertise in EBSD and *in situ* heating-cooling tests to help up in making these techniques usable to the community.

**5.2 Pending credits.** The FEI QUANTA 450 ESEM-FEG was acquired in 2016 by the University of Pisa through the “*Bando Grandi Attrezzature di Ateneo 2015*”. The latter entailed co-funding by individual faculty members. This economic support was then translated into laboratory hour credits. The costs of the renovation work carried out to set up the laboratory at *Palazzina Piaggio* sustained by DICI (26752 €) in 2016 were also translated into laboratory hour credits for that department. As of 2020.12.31, the pending credit situation is given in Table 2. In short, the laboratory has paid back ~46.6 k€ in credits over the four years of its activity, i.e. about 50% of the total ~99.7 k€.

**Table 2.** Laboratory credits (hours and equivalent value in € based the hourly fee with technical assistance of 50€) per department, as of 2020.12.31.

Department	Original economic support (€)	Credits (€)	Credits (hrs)
DICI	28500 <sup>a</sup> + 26752 <sup>b</sup>	14625 + 10810	509
DST	32000 <sup>a</sup>	19510	390
DCCI	9000 <sup>a</sup>	5200	104
DF	3500 <sup>a</sup>	2975	59.5
Grand total	99752	53120	1062.5

<sup>a</sup> Total individual faculty member co-funding.

<sup>b</sup> DICI support for renovation works.

**5.3 Income vs laboratory costs.** This year we had a significant decrease in hours worked. This was due to two factors: the ongoing pandemic and the breakage of the Bruker EDX detector. The safety restrictions imposed to contain the spread of COVID-19 have inevitably reduced the research and education work since early March 2020 (reduction of applications and staff), with the beginning of the application of the containment measures adopted by our government. The failure of the EDX detector then produced a reduction of the performance of the laboratory for three months (from end of July to end of October). This made the laboratory running only for imaging purposes, with the obvious further loss of users.

Altogether, the laboratory worked for 290 hours, equivalent to about 14.3 k€. This is about 2/5 of the minimum amount of work required to sustain lab costs. The latter include the Full-Risk maintenance annual contract of ~27573.76 € + IVA (thus 36640 € IVA inclusive) plus running costs of few k€ per year. Ideally the laboratory should work about 18 – 20 hours a week, assuming a reasonable work calendar of 40 weeks/year to produce an income of 36 – 40 k€. This is our objective for guaranteeing the service in the years to come.

The costs of the Full-Risk annual maintenance contract with the producer (THERMO) of ~34 k€, was covered by CISUP, contrary to previous years. The remote access system of ~3 k€ and gas lines of ~18 k€ were also covered by CISUP (“*fondi sicurezza/funzionamento*”).

The present balance of the ESEM-FEG FEI Quanta 450 CISUP account is 14.12 k€. Additional ~2 k€ of work carried out in 2020 are being cashed in.



**5.4 Maintenance contract extension.** Due the current pandemic, we obtained a 2-months extension of the Full Risk maintenance contract from THERMO. This implies that the instrument will be covered until 2025, November 28.

**5.5 Objectives.** Considering the dramatic effect of the pandemic coupled with the huge starting debt of the lab with co-founders (initially of 99.7 k€ and now reduced to 53.1 k€), the significant support of CISUP in covering this year maintenance contract fee has been vital for the ESEM-FEG FEI Quanta 450 laboratory. It is our top priority for the year 2021 to increase the performance of the laboratory despite the ongoing pandemic. Our specific objective is to bring the numbers of hours worked back to the sustainable figure of ~700 – 800 hours/year. This will be hopefully achieved through the remote access to the laboratory during the ongoing pandemic and by extending the usership to other departments (particularly life sciences and humanities).

**5.6. Cite our lab in your work.** Many of the papers which benefited of the work carried out in our FEI QUANTA 450 ESEM-FEG laboratory listed above do acknowledge our lab correctly in their methodology chapters (and acknowledgements) and we are most grateful to that. This is methodologically correct, ethically fair and vital for CISUP's health! However, many others do not (or not at all). This is something we should work on in the future. As also advertised in the laboratory web page, we invite our users to correctly cite our lab/instrument in their papers using – for instance - a sentence like the following one:

*“Scanning electron microscopy analyses were performed using a FEI QUANTA 450 ESEM-FEG at the Centro per la Integrazione della Strumentazione della Università di Pisa (CISUP)”*

Thanks!

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