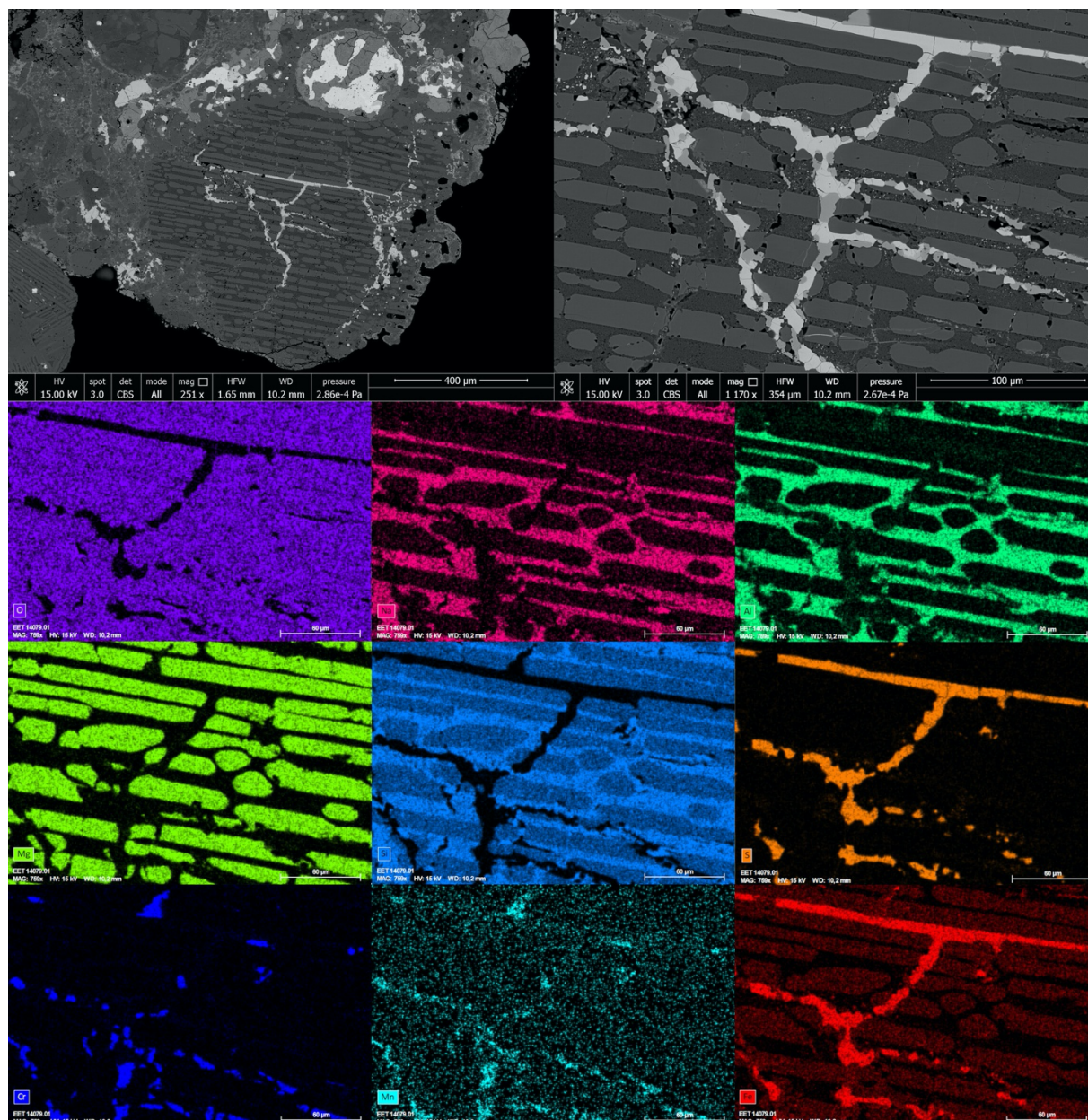


CISUP ESEM-FEG laboratory: 2021 activity report

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



Cover image: X-ray elemental map of barred-olivine chondrule in Antarctic chondrite EETA 14009. A) Courtesy of Anna Musolino

CISUP ESEM-FEG laboratory: 2021 activity report

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Abstract – This report serves to inform the UniPi community on the activities carried out by the CISUP ESEM-FEG laboratory during 2021, including: i) laboratory implementation; ii) hours worked; iii) user statistics; iv) research products; v) education and outreach; vi) issues and objectives. We practically managed to cover maintenance costs through laboratory work/paid machine time, despite a great deal of downtime due to prolonged vacuum problems. We also note our laboratory's growing impact on research and educational activities within and beyond UniPi, with the involvement of an increasing number of UniPi departments and external research institutes. The only down note is that the EBSD system was hardly used at all; its potential should be promoted, particularly amongst our PhD students and postdocs.

1. Laboratory implementation

MAPS software – In December 2021 we purchased the software MAPS for automated acquisition of high-resolution images from large areas. MAPS enables the navigation, tiling, stitching of image data. The area of interest is divided in multiple tiles which are recorded in a sequence and subsequently stitched to one single image. The size of the area is limited by the size of the stage rather than the image acquisition hardware. As a result, high resolution images of gigapixel size can be acquired. The system is configured to start the stitching process during image acquisition. Stitching accuracy is constantly monitored and in the unlikely event that the stitching quality confidence level is low, the software will highlight the issue enabling the user to do a manual alignment. Images can be stored in RAW, Tiff or HD View compatible format.

The software will be hopefully operative in spring 2022. Looking forward to working with it! Funding: CISUP.

2. Hours worked

Table 1 shows a breakdown of the hours worked by the ESEM-FEG laboratory since its establishment in fall 2016. During year 2021, the laboratory worked for 654 hours. This is slightly less than the average amount of hours (~700) worked during the three pre-pandemic years. As a result, the gross 2020 income is ~33 k€ against the 2017-19 average value of ~34.3 k€.

Table 1. ESEM-FEG usage (2021.12.31 update) in terms of hours (blue) and equivalent income (black).

Time period	Sep-Dec 2016		2017		2018		2019		2020		2021		Totals	
	hrs	€eq	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	483	24135	1781	89050
Experts (30 €/hr)	45.5	1365	188	5640	235	7043	239	7170	8	240	67	2010	715	21448
Non-institutional work (70 €/hr)									1	70	4	280	1	70
Other research institutes ^a							2	140			40	2800	2	140
External/private (100 €/hr)			35	3500	27	2700	38	3800	14	1400	34	3400	117	11700
Education											26	520		
<i>Pro bono</i>									9	0			9	0
Totals	111	4640	659	30940	820	37473	746	34435	290	14610	654	33045	3280	155153

Note that, in late June, we had a vacuum problem in the sample chamber which was solved by Thermo within the FULL RISK annual maintenance contract only in mid-September. As a result, unfortunately, we accumulated a great deal of downtime. On the bright side, we literally have all the electronics and pneumatic parts of the sample chamber vacuum system brand-new (Figure 1).



Figure 1. A moment of one of the several visits of THERMO engineers to solve the vacuum problem we had from late June to mid-September.

3. User statistics

The users of the ESEM-FEG laboratory over the year 2021 year include 48 research groups from 8 departments of the University of Pisa (DICI, DCCI, DST, DF, DII, DRT, DFARMA e Centro Piaggio) accounting for ~87% of the total workload; the remaining ~13% consisted of services for other research institutes (i.e., CNR), privates, educational activities and outreach. The department with the largest number of involved research groups is DICI (16) followed by DST (12), DCCI (11), DF (3), DII and Centro Piaggio (1), and DFARMA and DRT (1). The DST boasts the largest volume of laboratory work (~46% of the total), followed by DICI (20%), DCCI (13), DII and DF (2%), and DFARMA, DRT and Centro Piaggio (~1%). A welcome addition to the user's list is a visiting researcher from CEREGE, France, within the UniPi Visiting Fellows Programme.

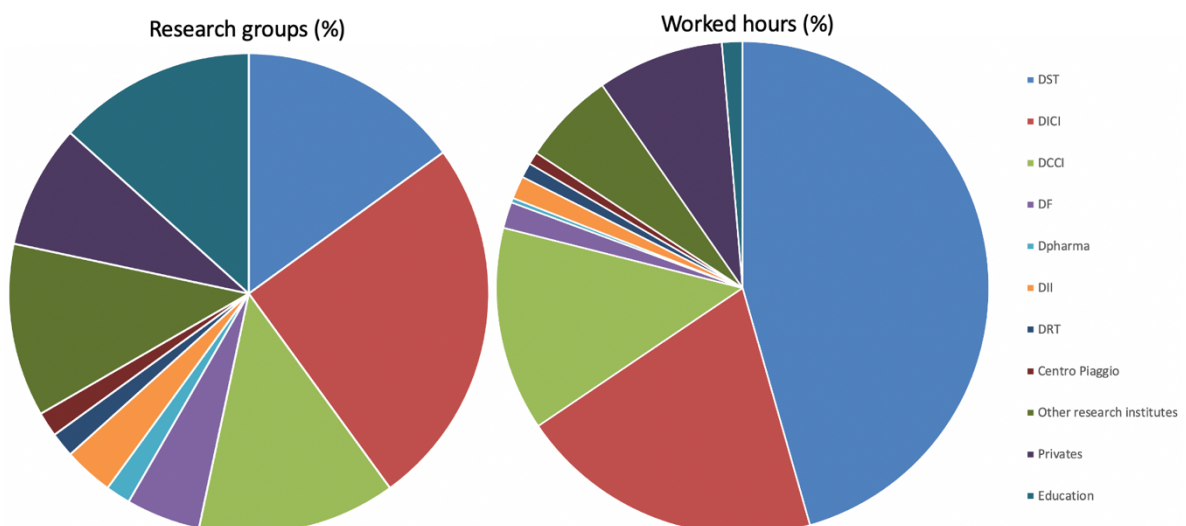


Figure 2. User statistics.

4. Research products

The database of the CISUP publications available at CISUP web-page <https://cisup.unipi.it/publications/> records **one book chapter** and **15 peer-reviewed papers** (see list below), including two published by *Nature Publishing Group*, produced by the above research groups. This result is remarkable, yet – at least quantitatively speaking – under par. Please double check your record in the CISUP database!

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- Catalano D. I. M., Conzatti L., Ilsouk M., Lahcini M., Manariti A., Maurina E., Raihane M., Rhouta B., Castelvetro V. (2021) Singling Out the Role of Molecular Weight in the Crystallization Kinetics of Polyester/Clay Bionanocomposites Obtained by In Situ Step Growth Polycondensation. *ACS Applied Polymer Materials* **3**, 5405-5415 <https://doi.org/10.1021/acsapm.1c00684>
- Chernozhukin S. M., González de Vega C., Artemieva N., Soens B., Belza J., Bolea-Fernandez E., Van Ginneken M., Glass B. P., Folco L., Genge M. J., Claeys Ph., Vanhaecke F., Goderis S. (2021) Isotopic evolution of planetary crusts by hypervelocity impacts evidenced by Fe in microtektites. *Nature Communication* **12**, 5646. <https://doi.org/10.1038/s41467-021-25819-6>
- Costa S., Fulignati P., Gioncada A., Pistolesi M, Bosch D., Bruguier O. (2021) Tracking metal evolution in arc magmas: Insights from the active volcano of La Fossa, Italy. *Lithos* 380–381, 105851, <https://doi.org/10.1016/j.lithos.2020.105851>.
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- Gigante V, Bosi L, Parlanti P, Gemmi M, Aliotta L, Lazzeri A. (2021) Analysis of the Damage Mechanism around the Crack Tip for Two Rubber-Toughened PLA-Based Blends. *Polymers*. 13(22):4053. <https://doi.org/10.3390/polym13224053>
- Gigante V, Cinelli P, Sandroni M, D'ambrosio R, Lazzeri A, Seggiani M. (2021) On the Use of Paper Sludge as Filler in Biocomposites for Injection Moulding. *Materials* **14**, 688. <https://doi.org/10.3390/ma14102688>
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5. Education and Outreach

We are proud to report that our lab has started to provide service for a number of educational activities, including practicals for MSc courses for DCCI, DST and DIC1 (~30 hours in total) as well as a short seminar (Scanning Electron Microscopy and Microanalyses for Geologists) for the Scuola di Dottorato Regionale di Scienze della Terra Pegaso.

On November 27th, our laboratory has actively participated to the BRIGHT-NIGHT la Notte Europea delle Ricercatrici e dei Ricercatori (BRIGHT 2021 project) with on-line show of our *wunderkammer* to the public. Many youngsters were fascinated by the imaging capabilities of our FEG-SEM. We showed high-tech materials, cosmic dust, volcanic hashes, minerals, fossils, but the real best seller of the day was inevitably a UniPi mosquito (Fig. 3).



Figure 3. The “star” of the BRIGHT 2021 event (...for ARAOs: found already dead, trapped in a spider web).

6. Issues and objectives

6.1 Performance. Like in the previous years, 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) and STEM system, but 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.

6.2 Income vs laboratory costs. This year we had a significant decrease in hours worked. This was mainly due vacuum problems in the sample chamber which produced a significant reduction of the performance of the laboratory for nearly three months (from end of June to mid-September) with continued disruption of the work program due to servicing.

Altogether, the laboratory worked for 654 hours, equivalent to 33045 €. This is about 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. This year we had no major running costs. We thus managed to nearly balance the lab costs. Surely downtime due the instrumental problems was a factor. Our objective for guaranteeing the ESEM-FEG lab service in the years to come is to work about 18 – 20 hours a week, assuming a reasonable work calendar of 40 weeks/year to produce a sustainable income of 36 – 40 k€.

6.3. Cite our lab in your work. 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. We strongly encourage to carry on with this far practice because beneficial to the entire CISUP community. Please check our laboratory web page, to correctly cite our lab/instrument in your 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!

***Acknowledgments** – Anna Musolino (INAF-IAPS Roma and formerly DST UniPi) is kindly acknowledged for making ESEM-FEG image available for this report's cover.*